VOL. XIV. 1908

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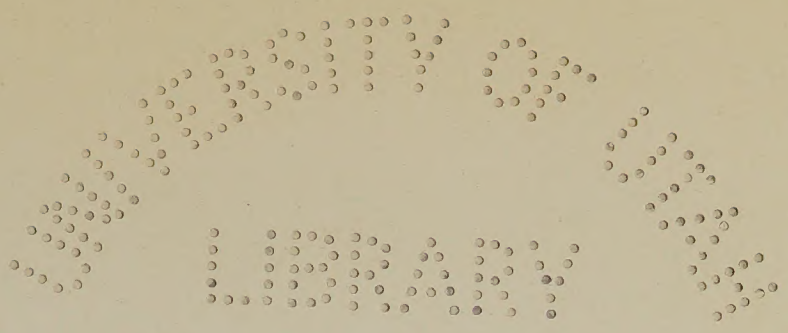
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TRANSACTIONS

OF THE

AMERICAN

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VOLUME XIV.

FOURTEENTH GENERAL MEETING

NEW YORK CITY, OCTOBER 30-31, 1908.

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VOLUME XIV. 1908.

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE FOURTEENTH GENERAL MEETING OF THE SOCIETY, HELD IN NEW YORK CITY, N. Y., OCTOBER 30-31, 1908.

(Number of members registered, 123; guests, 68; total, 191.)

SESSION OF OCTOBER 30th.

The morning session was called to order at 9.45 o'clock, in the Doremus Lecture Theatre of the Chemistry Building, College of the City of New York, New York City, President E. G. Acheson in the Chair.

Papers by A. Harold Porter and Francis C. Frary, of the University of Minnesota (read by Jos. W. Richards); E. P. Schoch, of the University of Texas (read by H. E. Patten); G. A. Hulett, of Princeton University; J. E. Mills, of the University of North Carolina (read by Jos. W. Richards); and G. W. Morden, of the University of Toronto, were read and discussed as printed in full in these Transactions.

During the morning session, President John H. Finley, of the College of the City of New York, was introduced to the Society by Prof. Charles Baskerville, and spoke in part:

"*Gentlemen of the Society:* We certainly have made very large provisions for the science of chemistry in this institution, this institution where in the past we have had Wolcott Gibbs, and

Dr. Doremus, and the younger Dr. Doremus, with whom I spent many hours in the discussion of the early plans of these buildings and these laboratories, and of Dr. Baskerville, Dr. Moody and Dr. Friedburg, and that splendid body of younger chemists who are associated with them. After we began work on this institution, we were obliged to raise the building in which we now are up one story, but it is still a distinct part, it has not yet dominated the other departments, but I am very glad to call your attention to the fact that though above ground it is quite a separate building, there is a subterranean passage connecting the department of chemistry with all the other departments—underneath we are all one, just as I suppose all the dominion of nature is a varied expression of the one truth. I am here not simply to welcome you to this department of the college presided over by Dr. Baskerville, but to the whole college, the college which is not only our college, but your college as well, the college of all who dwell within the boundaries of Greater New York. We extend you a most hearty welcome to this place, and we hope that your interest will abide in this institution, which is to be for this city what no other institution can be in its way. I am glad you are here, and hope you will make your stay as long as circumstances will allow." (Applause.)

The session then adjourned to the Main Building, where a luncheon was tendered by the Staff of the Department of Chemistry.

After luncheon a twenty-minute organ recital was given the Society by Prof. Samuel A. Baldwin, head of the Department of Music.

The meeting was again called to order at 2.05 P. M. by President Acheson.

Prof. Albert F. Ganz opened a general discussion on the "Corrosion of Iron," participated in by Messrs. Cushman, Toch, Gaines, Roberts, Bancroft, Hering, Sadtler and Sharp, as reported in full in the Transactions. Papers were read by A. A. Knudson, of New York; W. H. Walker, of Massachusetts Institute of Technology; W. D. Bancroft, of Cornell University; Maximilian Toch, of New York City, as printed with discussion in this volume. Prof. Charles Baskerville described the construction of the laboratory in which the meetings were held, and

of the educational system in use in the same, in a few well-chosen words.

SESSION OF OCTOBER 31st.

This session was called to order at 9.35, in the hall of the Chemists' Club, West Fifty-fifth Street, New York City, President Acheson in the Chair.

Papers by F. A. J. FitzGerald, Niagara Falls; W. McA. Johnson, Hartford; Carl Hering, Philadelphia; John Meyer, Philadelphia (read by C. A. Graves); E. A. Sperry, Brooklyn, were read and discussed as published in full in these Transactions.

The Committee of the Society, appointed to consider the "Conservation of our National Resources," through its Chairman, Mr. Edward R. Taylor, offered the following resolutions, which were unanimously adopted by the Society:

WHEREAS, at the conference of Governors which convened last May at the White House, at the instance of President Roosevelt, to consider the question of the conservation of our national resources, the necessity was emphasized of a closer relationship and a more intelligent co-operation among the unofficial associations which have an interest, in one way or another, in the conservation movement;

AND WHEREAS, in line with the sentiment expressed at the Governors Conference, there has been organized the Conservation League of America, whose officers are as follows: Theodore Roosevelt, honorary president; William J. Bryan and William H. Taft, honorary vice-presidents; Walter L. Fisher, president, and John Mitchell and Gustav H. Schwab, vice-presidents;

AND WHEREAS, the Conservation League of America has announced the following as its Statement of Principles;

WHEREAS, it is of the utmost importance that the natural resources of the nation shall be comprehensively and vigorously developed and utilized for the promotion of the public welfare, without waste, destruction, or needless impairment, and subject always to their intelligent conservation and the effective preservation of the rights and interests of the future generations of our people;

NOW THEREFORE, to secure the recognition and support of these principles by the people and by their representatives, we hereby unite in a National Conservation League, and adopt for ourselves the following, taken directly from the Declaration unanimously adopted by the Conference of Governors, convened by the President of the United States, in the White House, at "Washington, on May 13, 14 and 15, 1908:

"We do hereby declare the conviction that the great prosperity of our country rests upon the abundant resources of the land chosen by our forefathers for their homes, and where they laid the foundations of this great nation.

"We look upon these resources as a heritage to be made use of in establishing and promoting the comfort, prosperity and happiness of the American people, but not to be wasted, deteriorated or needlessly destroyed.

"We agree that our country's future is involved in this; that the great natural resources supply the material basis upon which our civilization must continue to depend and upon which the perpetuity of the nation itself rests.

"We agree that this material basis is threatened with exhaustion.

"We agree that the land should be so used that erosion and soil wash shall cease; and that there shall be reclamation of arid and semi-arid region by means of irrigation, and of swamp and overflowed regions by means of drainage; that the waters shall be so conserved and used as to promote navigation to enable the arid regions to be reclaimed by irrigation, and to develop power in the interests of the people; that the forests which regulate our rivers, support our industries, and promote the fertility and productiveness of the soil should be preserved and perpetuated; that the minerals found so abundantly beneath the surface should be so used as to prolong their utility; that the beauty, healthfulness and habitability of our country should be preserved and increased; that sources of national wealth exist for the benefit of the people, and that monopoly thereof should not be tolerated.

"We declare our firm conviction that this conservation of our natural resources is a subject of transcendent importance, which should engage unremittingly the attention of the nation, the States, and the people in earnest co-operation.

"We agree that this co-operation should find expression in suitable action by the Congress and by the Legislatures of the several States.

"Let us conserve the foundations of our prosperity."

And Whereas, the political and industrial non-partisanship of the Conservation League of America is insured by the Statement of Principles, the representative character of its officers, and especially by the broad scope of its membership of unofficial organizations vitally concerned with every phase of the Conservation movement; therefore,

Be It Resolved, by the American Electrochemical Society, in convention assembled, that we express our unqualified approval of the objects and purposes of the Conservation League of America, and pledge it our active support in the patriotic and unselfish work it has undertaken.

And be it Further Resolved, that our Secretary be instructed to send a copy of these resolutions to the Conservation League of America, with the request that the League advise us what, if anything, this organization can do to assist in this important work.

Mr. Gifford Pinchot,

President Conservation Commission:

The American Electrochemical Society, feeling deep interest in the conservation of all the resources of our country, and being gratified that so important a work is under your direction, respectfully suggests:

That the excellent work determined upon by the Forestry Department be thoroughly supplemented by water storage in natural and artificial lakes and reservoirs, according to Bulletins 143 and 144, for diminishing of floods, and the increase of water power and navigation.

We specifically recommend the acquirement by the General Government of the Appalachian, Adirondacks and White Mountains Head Waters for the above purposes.

We deprecate such development of forests that shall leave them in a wild state under such restrictions as that shall forbid their use for timber, etc., as it becomes ripe and in best condition for use.

We also deprecate such restrictions in forest reserves concerning the enlargement of lake and the creation of artificial reservoirs as are necessary for the best and largest possible conservation of water for the purposes of power, restriction of floods and the highest development of navigation in our rivers and canals.

We deprecate the construction of a barge canal through the great State of New York that makes no provision for the development of water power in this electrical age, not even for the haulage of the boats to be operated thereon. And we urge the General Government to investigate and, if found practicable, take in hand the construction of a navigation and power canal that shall meet the great needs of the commerce of the great and growing West. And that shall develop the manufacturing interests dependent upon cheap water power combined with cheap water transportation commensurate with the magnificent resources involved.

We respectfully urge the co-ordination and harmonious development of all of our resources, recognizing that no one department has all there is of good, but that each should supplement the other for the best good of our whole people.

We respectfully urge a broad view of the great value the higher and cheaper lands of the water-sheds previously referred to have in their reservoir, storage capacity to hold back floods from the comparatively speaking, millions of acres of lands at lower levels now annually inundated.

Resolved, that in the important functions you have undertaken we accord to you our most hearty support and, so far as we be able, assistance.

Resolved, that a copy of these resolutions be forwarded to the Conservation Commission by the Secretary of our Society.

Respectfully Submitted.

E. R. Taylor,
C. A. Doremus,
W. D. Bancroft.

On motion of Mr. Carl Hering, a vote of thanks was ordered conveyed to The College of the City of New York; the Staff of the Department of Chemistry of the College of the City of

New York; The Chemists' Club; The Balbach Smelting Works; the Local Committee of the Society; for their interest and aid in making successful the Fourteenth General Meeting of the Society.

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Crider, J. S.
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Salom, P. G.
Schamberg, M.
Smith, E. F.
Smith, E. W.
Smith, W.
Snook, H. C.
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Voge, A. L.

A paper read by Jos. W. Richards at the Fourteenth General Meeting of the American Electrochemical Society, in New York City, October 30-31, 1908; President E. G. Acheson in the Chair.

CHEMICAL ENERGY.

By J. E. Mills.

If 16 grams of oxygen gas at 0° C are placed in contact with 2.016 grams of hydrogen gas at the same temperature, and both under atmospheric pressure, no reaction is evident. But if a negligible amount of heat be supplied to the mixture in the form of an electric spark, a violent reaction takes place, and 18.016 grams of water are formed and 68,511* calories of heat are given out. If the reaction took place at a different temperature, or under different conditions of pressure or volume, the amount of heat evolved would have been different, but the chemical reaction in each case would have been the same.

From whence came the enormous amount of heat given out by the reaction? Why and how does the amount of heat given out vary under different conditions? Why did not the reaction take place when the hydrogen and oxygen were first mixed?

It is the purpose of this paper to answer these questions and to point out certain facts concerning chemical energy and certain possibilities regarding the relation of chemical energy to electrical and other forms of energy.

THE SOURCE OF THE ENERGY GIVEN OUT BY THE REACTION.

If the term "absolute zero of temperature" be used, some one will object that there may be no absolute zero of temperature, that we are not certain of the existence of molecules, and that if they do exist we certainly do not know that they cease to move at the so-called absolute zero. Granting for the present the validity of the objection, we will not use the term absolute zero,

* Mixter, Amer. Jour. of Science [4], **16**, 214 (1903). The value given is the mean of the values obtained by Thomsen, Schüller and Wartha, Than, and Mixter, after reduction to the calorie at 15° to 16° as the unit. Mixter thinks the value correct to one-tenth of one per cent.

but will speak of — 273° C. No one will deny the existence of that temperature since a temperature below — 260° C has already been reached.

For reasons which will appear later I wish to get the total amount of heat necessary to raise 2.016 grams of hydrogen, and 16 grams of oxygen, and 18.016 grams of water (ice), from — 273° C to 0° C. The measurements given in the table below enable this data to be obtained correct to within a few calories.

	Melting Point	Boiling Point	Specific Heat of Solid	Specific Heat of Liquid	Heat of Fusion	Heat of Vaporization	Specific Heat of Gas
Hydrogen	—258.9[1]	—252.59[1]	2.3[2]	3 4[3]	16[3]	123.1[3]	3 410[4]
Oxygen	—227[3]	—182.80[1]	0.25[2]	0.347[5]	. .	50 97[5]	0 2 75[6]
Water	0	100	0.294[3]	1 000	79.90[7]		

The total heat necessary to raise these substances from — 273° to 0° C may therefore be estimated as follows:

Hydrogen (1 gram).

Specific heat of solid, —273° to —258.9° = 14.1 × 2.3.. =	32.4	calories
Latent heat of fusion ..... =	16	"
Specific heat of liquid, —258.9° to —252.59° = 6.31 × 3.4 =	21.4	"
Latent heat of vaporization ..... =	123.1	"
Specific heat of gas, —252.59 to 0° = 252.59 × 3.410.. =	861.3	"
Total heat, —273° to 0°C for 1 gram..... =	1,054.2	"
Total heat, —273° to 0°C for 2.016 grams..... =	2,125.2	"

Oxygen (1 gram).

Specific heat of solid, —273° to —227° = 46 × 0.25.. =	11.50	calories
Latent heat of fusion, estimated..... =	5.70	"
Specific heat of liquid, —227° to —182.8° = 44.2 × 0.347. =	15.34	"
Latent heat of vaporization..... =	50.97	"
Specific heat of gas, —182.8° to 0° = 182.8 × 0.2175.. =	39.75	"
Total heat, —273° to 0°C for 1 gram..... =	123.26	"
Total heat, —273° to 0°C for 16 grams..... =	1,972.2	"

[1] Travers, Senter and Jaquerod, Phil. Trans., **200**A, 170 (1903).

[2] This value was estimated by Kopp from the specific heat of its solid compounds. We know almost with certainty that the specific heat of the solid is not so great as the specific heat of the liquid, and probably the value given is nearly correct.

[3] Dewar, Proc. Roy. Soc., **76**A, 325 (1905); **69**, 361 (1902); **73**, 251 (1904). He determined the specific heat of ice, —252.6 to —188 = 0.146, —78 to —188 = 0.285, —78 to —18° = 0.463.

[4] Wiedemann, Pogg. Ann., **157**, 1 (1876); Phil. Mag [5] **2**, 81 (1876).

[5] Alt, Zeit, Phys. Chem., **51**, 252 (1905); Phys. Zeits., **6**, 346 (1905); Ann der Physik, **13**, 1010 (1904); Dissertation, München, 1903.

[6] Regnault, Mem. de l' Acad., **26**, 1 (1862).

[7] Smith, Phys. Rev., **17**, 193 (1903).

Water (1 gram).

Specific heat of solid, 273° to 0°	=	80.34 calories
Latent heat of fusion	=	79.90 "
Total heat, —273° to 0°C for 1 gram	=	160.24 "
Total heat, —273° to 0°C for 18.016 grams	=	2,886.9 "

The 18.016 grams of water at 0° C contain, therefore, at least 2,887 calories, and since 68,511 calories of heat were given out by the reaction of the hydrogen and oxygen gases, these gases before the reaction contained at least 71,398 calories of energy. Of this total amount of energy, 2,125 calories were supplied to the 2.016 grams of hydrogen, and 1,972 calories were supplied to the 16 grams of oxygen, in converting them from the solid condition at — 273° C to the gaseous condition at 0° C — a total of 4,097 calories. It follows, therefore, that while in the solid condition at — 273° C the oxygen and hydrogen contained at least 71,398 — 4,097 = 67,301 calories of energy. Now Dewar[8] has shown, by slight extrapolation of measurements extending to — 258.9° C that the molecular volume of oxygen at — 273° C is 21.21 c.c., of hydrogen 24.18 c.c., and of ice 19.21 c.c.

Therefore the 16 grams of oxygen and the 2.016 grams of hydrogen together at — 273 ° C occupy 34.78 c.c., and when occupying this volume at that temperature they contain 67,300 calories more of energy than does the 18.016 grams of ice which they form if united, and which occupies a volume of 19.21 c.c.

There is no supposition whatever connected with this fact, except the slight uncertainty due to the measurements and their extrapolation, and this uncertainty is probably not greater than 100 calories. A study of substances with higher melting points confirms the view that the errors introduced by the extrapolation are very slight, and we feel confident in the practical accuracy of our statement.

The total energy possessed by the hydrogen and oxygen at — 273° C is probably very much more than 67,300 calories, and may be many times that amount, for we have no evidence whatever as to the energy which the ice possesses at that temperature. We only know that the energy of the ice is 67,300 calories less than the amount possessed by the equivalent hydrogen and oxy-

[8] Dewar, Proc. Roy. Soc., **73**, 251 (1904).

gen before the combination. The ice may, and in our opinion certainly does, possess a vast amount of energy. Its store of energy is perhaps comparable to that possessed by radium.

In what form does the 67,300 calories of energy possessed by the 34.78 c.c. of hydrogen and oxygen at — 273° C exist? The answer to the question, "What is energy?" and "What is matter?" must both be given before any sufficient answer to the first question could be made. But there is certainly no harm in stating what we do know concerning the answer to that question, and it is this:

If the same mechanical laws which govern larger masses of matter and energy at higher temperatures apply to the hydrogen and oxygen at — 273° C, then a stable system could not exist if all of the energy were kinetic, or if all of the energy were potential,[9] *but some portion of the energy must be potential and some portion must be kinetic.*

There has never been the slightest evidence produced to show that these mechanical laws do cease to hold at — 273° C, or with small subdivisions of mass, and therefore we think it probable that at — 273° C the small particles—atoms if you please to call them so—of hydrogen and oxygen, are in exceedingly rapid motion, and are held together by some force which gives rise to a potential energy. The force we will call chemical affinity, and the energy we will call chemical energy.

It is not out of place to point out here, for it is very suggestive, that if we have two masses of matter which attract each other inversely as the square of their distance apart, and which form a stable system, they will revolve around their common center of gravity, and *they cannot give out a greater amount of energy than they retain as kinetic energy.* Just what relation would hold if we had numerous particles governed by the same laws, mathematics has not yet proved adequate to reveal. The relation may be a very simple one in spite of the mathematical complexity of the problem.

The answer to our first question is, therefore, that 67,300 calories of the total energy possessed by the 2.016 grams of hydrogen and the 16 grams of oxygen at 0° C existed in some

[9] See Meyer, Kinetic Theory of Gases, p. 344, English translation of second revised edition.

form in those substances at — 273° C, and 4,097 calories of energy were added in raising those substances to 0° C and changing them to the gaseous condition.

ENERGY CHANGES CAUSED BY A RISE IN TEMPERATURE.

We wish next to show why 4,097 calories of energy were necessary to change the temperature and condition of the hydrogen and oxygen as described.

Of this amount of energy 2,125 calories were added to the hydrogen. Now the hydrogen at — 273° C originally occupied a volume of 24.18 c.c., and at 0° C it occupied a volume of 22,431 c.c. This expansion necessitated the expenditure of work in pushing back the atmosphere. The work so done we will call $E\epsilon$, and it may be calculated for one gram from the equation:

$$(1). \qquad E\epsilon = 0.000031833\ P\ (V - v)\ ^{10} \text{ calories},$$

where P is the pressure in millimeters of mercury, and V is the final and v the original volume. For hydrogen, therefore, the external work performed during the expansion is $0.000031833 \times 760\ (11{,}126.4 - 12.1)\ 2.016 = 542.1$ calories.

During the expansion of the hydrogen the molecules of the hydrogen were moved further apart, and a certain amount of energy was necessary to overcome the attraction between the hydrogen molecules. This amount of energy, which we will denote by Ea, may be calculated, as the author has shown in a series of papers,[11] from the equation:

$$(2). \qquad Ea = \mu' (\sqrt[3]{d} - \sqrt[3]{D}) \text{ calories}.$$

where d and D are the density in the original and final conditions respectively, and μ' is a constant which may be obtained from the following equation:

[10] The constants used in this paper are:
Density of mercury = 13.5956 at 0° referred to water at 4°C.
Attraction of gravity = 980.5966 at latitude 45° and sea level.
Atomic weight of oxygen = 16.00.
Density of hydrogen at latitude 45° and sea level = 0.0000898765.
This is an average of the values given by Morley, Smithsonian Contributions to Knowledge, No. 980, and the value given by Rayleigh, Proc. Roy. Soc., **53**, No. 322, 147, after reduction to latitude 45°. Rayleigh's value was an average of his own value, that of Leduc, and the value of Regnault as corrected by Crafts.
The unit calorie is taken as from 15° to 16°C and as equal to 41,880,000 ergs. This is Rowland's value as corrected by Day, Phys. Rev., **6**, 194 (1898). See also Ber. dtsch. Physik. Ges., **6**, p, 578.

[11] Jour. Phys. Chem., **6** 209 (1902); **8**, 383 (1904); **8**, 593 (1904); **9**, 402 (1905); **10**, 1 (1906); **11**, 132 (1907); **11**, 594 (1907). The equation has been proved to hold for liquids as they expand to the volume of the saturated vapor. The author believes that the equation, which was theoretically derived, may be applied as above, and the result appears to justify the belief.

$$(3). \qquad \frac{L - E\epsilon}{\sqrt[3]{d} - \sqrt[3]{D}} = \mu'$$

Here L is the heat of vaporization, $E\epsilon$ is the energy spent in pushing back the atmosphere during the vaporization, and is calculated from equation (1), d is the density of the liquid, and D the density of the saturated vapor at the temperature of vaporization.

To calculate μ' for hydrogen, L = 123.1 calories. The density of the liquid at its boiling point has been determined by Dewar as 0.070025, corresponding to a volume of 14.28 c.c. per gram. The density of the saturated vapor may be calculated on the supposition that it obeys the gas laws, the error so introduced being negligible. The equation for this calculation is:

$$(4). \qquad D = \frac{.00001601_4 \, Pm}{T}$$

Since T is 20.41°, D becomes 0.001202, and the corresponding volume is 831.9 c.c. From equation (1), $E\epsilon$ is found to be 19.78 calories, and $L - E\epsilon$ is therefore 103.32. Dividing this by the value of $\sqrt[3]{d} - \sqrt[3]{D}$, namely, 0.3059, we have for μ' the value 337.7. Substituting this value in equation (2), and using for d the density of hydrogen at — 273° C as given by Dewar, 0.08272, and for D the value already given at 0° under standard conditions, namely 0.00008988, we have finally,

$$E\alpha = 337.7 \times .3909 \times 2.016 = 266.1 \text{ calories.}$$

It is not necessary to pause here to defend the kinetic theory of gases. That theory represents the attempt to extend the mechanical laws which govern large masses of matter to smaller particles of the same material. Its foundation upon these mechanical laws is quite as secure as are the foundations of any system of thermodynamics yet advanced—both theories alike resting upon observed laws to which no single exception has ever been found. Moreover it has led to important discoveries and correllated numerous facts.

Making use now of the kinetic theory, it is easy to calculate the energy necessary to add to the gram molecular weight of hydrogen in order that its molecules should have at 0° C sufficient motion to produce the pressure which we know is produced

at that temperature. Using the constants which we have adopted, and calling this kinetic energy E_k, the equation takes the form:

$$(5). \qquad E_k = \frac{3\ R\ T}{2} = \frac{2.9817\ T}{m} \text{ calories.}$$

Whence the kinetic energy of the gram molecular weight of hydrogen at 0° C is 814.0 calories.

We know also from the kinetic theory of gases that whenever we increase the kinetic energy of a molecule we at the same time increase its internal energy, which we will call E_I. The increase in the internal energy is proportional to the increase of the kinetic energy, and its amount may be calculated from the relation first proposed by Waterston, namely,

$$(6). \qquad \gamma = \frac{\text{specific heat of gas at constant pressure}}{\text{specific heat of gas at constant volume}} =$$

$$\frac{Sp}{Sv} = \frac{3/2\ R + E_I + R}{3/2\ R + E_I} = \frac{5/2\ R + E_I}{3/2\ R + E_I}$$

Now putting in the place of R its value $2/3\ \frac{E_k}{T}$, and solving for E_I, we obtain finally, since T is equal to 1;

$$(7). \qquad E_I = \frac{5/3 - \gamma}{\gamma - 1} E_k \text{ calories.}$$

For hydrogen $\gamma = 1.4084$ from the measurements of Lummer and Pringsheim,[12] and so we have for E_I for 2.016 grams of hydrogen, 514.6 calories.

As a summary, therefore, we have for the total energy necessary to change 2.016 grams of hydrogen from the solid condition at — 273° C to the gaseous condition at 0° C under 760 mms pressure:

F_ϵ	= Energy necessary to overcome external pressure =	542.1	calories
E_a	= Energy necessary to overcome molecular attraction =	266.1	"
E_k	= Energy necessary for translational motion..... =	814.0	"
E_I	= Energy necessary for internal work..... =	514.6	"
	Total ..... =	2,136.8	"

[12] Smithsonian Contributions to Knowledge, No. 1126.

Now, as a matter of observation, we found that 2,125 calories were added to effect the above-mentioned change in the condition of the hydrogen, and the agreement is certainly within the limit of experimental errors involved in the measurements.

An exactly similar calculation regarding the energy added to the oxygen can be made from the following data:

Density of solid at —273°C.	=	1.5154[1]	
Density of liquid at boiling point.	=	1.134[2]	
Density of vapor at its boiling point.	=	0.00440[3]	
Density of oxygen at 0°C.	=	0.001429[4]	
Boiling point of oxygen.	=	—182.8°C[5]	
γ = Ratio of specific heats of this gas.	=	1.4025[6]	
Heat of vaporization of oxygen at boiling point...	=	50.97	calories[7]
From the above $L - E_\epsilon = 45.49$ calories, and μ'	=	51.74	

We have finally for the energy necessary to change one gram of oxygen from the solid condition at — 273° C to the gaseous condition at 0° C and under 760 mms. pressure:

E_ϵ = Energy necessary to overcome external pressure..	=	16.89	calories
E_a = Energy necessary to overcome molecular attraction	=	53.62	"
E_k = Energy necessary for translational motion.	=	25.44	"
E_I = Energy necessary for internal work.	=	16.69	"
Total	=	112.64	"

To effect the above-mentioned change for 16 grams of oxygen will require 1,802 calories. The amount actually added to the oxygen was found to be 1,972 calories.

The divergence here, amounting to about 10 calories per gram, cannot, we believe, be considered as entirely due to experimental error, and we have made an extended investigation as to its cause. As a result of this investigation, the details of which it would be impossible to reproduce within the limits of this paper, we believe that besides the so-called external, attractive, kinetic and internal energy, for which allowance has been made, *all* bodies, while in the solid condition require yet an additional

[1] Dewar, Proc. Roy. Soc., **73**, 251 (1904).
[2] Ladenburg and Krügel, Ber. d. deutsch. Chem. Ges., **32**, 1415 (1899).
[3] Dewar, Proc. Roy. Soc., **73**, 255 (1904).
[4] For authorities, see note on the density of hydrogen p. 5.
[5] See Note 1, Table I.
[6] Müller, Dissertation, Breslau, 1882.
[7] See Note 5, Table I.

amount of energy, the exact office of which we are unable to explain. The amount of this unaccounted for energy varies with the absolute temperature of the melting point of the body. In the case of hydrogen because of its very low melting point, 14° absolute, the amount thus unaccounted for was well within the limit of experimental error. But with oxygen this is not the case.

We are led here to add that the total energy necessary to change a solid monatomic element from — 273° to the liquid condition at its melting point, appears to us to be three times the kinetic energy of translation required by the element at that temperature; or, in other words, 8.94 T calories, where T is the absolute temperature of the melting point. Prof. J. W. Richards[13] has reached nearly the same conclusion, considering the total heat in the molten metal at its melting point to be 10 T calories. We will publish our data and conclusions upon this subject later.

Unfortunately our calculations cannot be extended to water, for it is an associated substance.

Except for the discrepancy of 170 calories, we have shown why and how the 4,097 calories of energy added to the hydrogen and oxygen to change them from their condition at — 273° C to their condition at 0° C were expended, and have, therefore, in part at least, answered the second question as to why and how the amount of heat given out by the reaction of the hydrogen and oxygen will vary under different conditions of volume, temperature and pressure.

THE CHEMICAL CHANGES INVOLVED IN THE REACTION.

The answer to the third question as to why the reaction did not take place when the hydrogen and oxygen were first mixed, and before the passage of the electric spark, has doubtless occurred to every one. It was first necessary to loosen the union of some of the hydrogen atoms from their combination with other hydrogen atoms, and the union of some of the oxygen atoms from their combination with other oxygen atoms. Then once free, the hydrogen atoms and the oxygen atoms unite, and in so doing give out enough heat to loosen the neighboring

[13] Trans. Amer. Electrochem. Soc., **13**, 447 (1908).

hydrogen and oxygen molecules, and these in their turn unite, and thus the reaction is propagated.

But it is perhaps not so clearly recognized, that the force which we call chemical affinity, is probably never directly affected by a rise in temperature. Certainly in the case under consideration we have accounted for all of the energy added to the hydrogen and the oxygen from — 273° to 0° C, save 170 calories, as having been used to effect external work, overcome molecular attraction, and effect an increase in the translational and internal motion of the molecules, and the original 67,300 calories possessed at — 273° C appears to have been held, intact as it were, neither increased nor diminished, by the changes of temperature, pressure, and volume necessary to raise the hydrogen and oxygen to their condition at 0° C.

The 67,300 calories represents the total energy change of the three chemical reactions,

$$H_2 = H + H,$$
$$1/2O_2 = 1/2\ (O + O),$$
$$H + H + O = H_2O,$$

when taking place at — 273° C.

We cannot at present determine how much energy would be required by each reaction if it alone took place, but when a sufficient number of well-chosen chemical reactions have been thus studied it may be possible to find the key to all, and to determine not alone the force which acts between any two atoms, but the law which governs the distance apart of the atoms as well, and the exact amount of energy which will be given out by the different chemical and physical changes involved. A study of the energy changes accompanying, and the conditions necessary for, the dissociation of simple molecules should greatly aid in the solution of the problem.

CONCLUSIONS SUGGESTED REGARDING CHEMICAL ENERGY.

The study of chemical energy as set forth in this paper has covered but a small portion of the field. The electron theory, the valency theory of Barlow and Pope,[14] the work of Traube[15]

[14] Jour. Chem. Soc., **89**, 1675 (1906); **91**, 1150 (1907).

[15] Numerous papers. Among others, his Grundriss d. phys. Chemie; Boltzmann: Festschrift (1904); Zeit. Anorg. Chemie, **40**, 380 (1904); Sammlung, Chemischer und chemisch-technischer Vorträge, **4**, 255; Berichte, **40**, 130, 723, 734, 2341 (1907.)

upon atomic volumes, of T. W. Richards[16] upon the compressibility of the elements and the relations of volume to chemical energy, and of Werner[17] upon the conductivity and valency relations of certain classes of organic compounds, all constitute recent notable advances in our knowledge of chemical force and its action. I make no attempt to consider the entire field. In my own study of molecular and chemical attraction I have been most forcibly impressed by the possible truth of three ideas:

First.—*That the attractive forces, whatever their nature, whether chemical, molecular, magnetic, electrical, or gravitational, which proceed from a particle are definite in amount. If this attraction is exerted upon another particle the amount of the attraction remaining to be exerted upon other particles is diminished by an exactly equivalent amount.*

Second.—In the general resemblance between all of the attractive forces as shown by the following summary:

Force	Medium of Propagation	Effect of Temperature	Is the Attraction Neutralized?	Is the Attraction Directive?	Law of Distance, d	Numerator Factor of Force
Chemical	Ether	No Effect	Neutralized	Yes	?	Nature of Atom × Nature of Atom
Molecular	"	No Effect	Neutralized	?	$\frac{1}{d^2}$	Nature of Molecule × Nature of Molecule
Magnetic	"	?	Neutralized	Yes	$\frac{1}{d^2}$	Strength of Pole × Strength of Pole
Electrical	"	?	Neutralized	Yes	$\frac{1}{d^2}$	Charge × Charge
Gravitational	"	No Effect	Not Neutralized	No	$\frac{1}{d^2}$	Mass × Mass

This table I have given, and to some extent discussed in a previous paper,[18] and I have there suggested that possibly all of the attractive forces in reality obey the same law, and that the apparent exceptions are caused by our incomplete knowledge of

[16] Proc. Amer. Acad., **37**, 3 (1901); **37**, 399 (1901); **38**, 293 (1902); **39**, 581 (1904). The Compressibilities of the Elements and Their Periodic Relations.

[17] Berichte, **40**, 15 (1907), and numerous other papers.

[18] Jour. Phys. Chem., **11**, 157 (1907).

the facts and a misunderstanding of their true relationship. Thus Newton thought that molecular attraction could not be explained as due to gravitational attraction acting between the extremely small molecular distances, because the gravitational force was by no means great enough to account for the molecular cohesion observed, and because the molecular cohesion diminished too rapidly with the distance apart of the molecules. Yet as evidence against this view the author has shown[19] that the equation deduced by Helmholtz to represent the energy given out by the sun on contraction under the influence of gravitation, can be used to accurately calculate the energy given out by a substance in changing from the gaseous to the liquid condition, provided only that the constant of Helmholtz's equation be changed.

Newton did not consider that the total available attraction of a molecule was diminished by its attraction for other molecules. In effect Newton's idea makes both molecular and gravitational attraction infinite in amount, or at least capable of infinite multiplication by the introduction of new particles. If the attractive forces proceeding from a particle are definite in amount, then it is possible, I think, that the molecular attraction is merely the residual chemical attraction, and gravitational attraction is the residual molecular attraction. Magnetic force is possibly a manifestation of molecular attraction, and electrical force is possibly identical with chemical force.

In the paper cited above, we have discussed this idea somewhat more in detail, and have there suggested that possibly all of the attractive forces could be measured by

$$\frac{\text{amount of attraction neutralized at unit distance} \times \text{amount of attraction neutralized at unit distance}}{d^2}$$

where d is the distance apart of the attracting particles.

The view which we have expressed above enables us to obtain a very clear idea of the theoretical significance of temperature and the absolute zero. If the atoms of a substance be supposed at an infinite distance apart, the total energy possessed by an atom may be of two kinds. First, the potential energy of *its*

[19] Jour. Phys. Chem., **11**, 145 to 153 (1907).

own attractive force, and second, a possible energy of translational motion. This latter motion, under the conditions named, would be a measure of the temperature of the body. Now as the atoms move closer together, the potential energy of attraction is partially transformed into translational motion, but this additional motion of the atom so gained is not entirely motion which constitutes temperature. Of the total gain of kinetic energy caused by the attraction some must be retained for stable equilibrium under the conditions, as a balance, as it were, for the attractive force. (The amount so retained can be calculated, where the law of the force is known, for the simplest case of two particles). Only the excess of energy above this retained amount becomes visible as temperature.

The temperature motion is the excess of translational motion impressed upon an atom or molecule beyond that which it would retain as a necessary attachment of its potential energy under the conditions considered.

Thus at — 273° C we undoubtedly, I think, have atomic motion, and might have molecular motion, but to my mind at least the extreme theoretical and practical importance of that point as the "absolute zero of temperature" is not decreased thereby.

Third.—Regarding more particularly the relationship between electrical and chemical forces, I have in my own mind connected the power to conduct electricity, whether the substance is in the solid, liquid, or gaseous condition, not primarily with molecules, nor with ions, nor even perhaps with electrons. The essential requisite appears to me to be a free, that is, an unabsorbed attraction. My meaning will become clear by a brief discussion.

The best conductors in the solid condition are the metals such as copper, silver, platinum, etc., and these metals are monatomic. That is to say, the chemical attraction which the atoms of these metals possess is not bound hard and fast to another atom, a mutual absorption of the attraction between the two atoms having taken place. The attraction is indeed to some extent absorbed by the neighboring atoms, for I think there is no very clear distinction to be drawn between molecular and chemical attraction, but the attraction is so distributed that it is

easily loosened and becomes free. In the metals which conduct less well, such as bismuth or antimony, the atoms, as is well known, are partly combined, and the attraction is therefore more difficult to free, and it becomes more difficult to propagate the attractive wave. Our best insulators are the most stable chemical compounds, glass, clay, oxides, salts, etc., or compounds such as rubber, where the chemical attraction of the atoms is almost completely saturated by other definite atoms. If these bodies are to conduct electricity the stable chemical union must be broken down, or at least loosened. This loosening of the chemical union happens usually with an increase of temperature.

A stable gas, such as oxygen or hydrogen, is a poor conductor. If such a gas is to be made a conductor it must be "ionized," that is, the chemical attraction which before was definitely absorbed by some other atom must be made free.

Considering solutions, an acid, base, or salt, when dissolved in water becomes a good conductor, because, using sodium chloride as an illustration, the condition in the solid is represented by a sodium atom and a chlorine atom which mutually saturate each other's attraction. As a disssolved salt, the sodium attraction is partially or wholly loosened from the chlorine, and this attraction distributes itself upon the hydroxyl portions of the neighboring water molecules. The attraction of the chlorine for the sodium, is, of course, loosened at the same time, and distributes itself upon the hydrogen of the neighboring molecules. Whether the water molecules and the sodium chloride molecules are definitely pulled apart I do not know, but the facts of the electrolytic dissociation theory form partial evidence that they are. In the case of acids, bases, and salts, the breaking apart of the dissolved molecule is possibly complete at "infinite dilution." In other cases the dissolved substance retains its chemical identity, and this means nothing else than that the mutual atomic attractions remain definitely bound, and the substance remains a poor conductor.

Unless a distributed chemical affinity is equivalent to an electrical charge I see no reason for supposing that "ions" have electrical charges. Otherwise I think that the electrolytic dissociation theory represents an essential part of the truth. But no less essential is the point so repeatedly emphasized by Kahlen-

berg, that there is a union between the solvent and the dissolved substance. We believe this union to be stronger or more complete the more completely "dissociated" is the dissolved substance. The hydrate theory, the dragging of the solvent molecules by the ions, and the change in concentration around the electrodes, bear decidedly upon this point. Kohlrausch[20] advances essentially the same idea, and it is further discussed and supported by Bousfield.[21] Lewis[22] in his review of recent advances in physical chemistry, says, "In many investigations it has been shown that the solutes which give conducting solutions are able to form definite solid compounds with the solvent, thus indicating that in the solution, too, there is a union of solvent and solute."

We have only extended these ideas a little by saying that atoms whose lines of force radiate out indiscriminately to other atoms are good conductors. Atoms whose lines of attractive force extend to certain other definite atoms, the attraction of each being mutually saturated thereby, are very poor conductors.

University of North Carolina.
Chapel Hill, N. C.
October 2, 1908.

[20] Proc. Roy. Soc., **71**, 338 (1903).
[21] Zeit. Phys. Chemie, **53**, 257 (1905).
[22] Jour. Amer. Chem. Soc., **28**, 906 (1906).

A paper read by Jos. W. Richards at the Fourteenth General Meeting of the American Electrochemical Society, in New York City, October 30, 1908; President E. G. Acheson in the Chair.

THE USE OF A MERCURY CATHODE IN THE DETERMINATION OF METALS.

By A. Harold Porter and Francis C. Frary.

Ever since Gibbs[1] first proposed the use of the mercury cathode for the electroanalytical determination of the metals, it has until recently been generally assumed that the method was inherently accurate, and that the principal care necessary was to effect complete precipitation of the metal from the solution. Recently, however, Price[2] has investigated the precipitation of zinc upon a mercury cathode with a rotating anode, and shown, contrary to previous statements by others,[3] that the results are uniformly low. An examination of the results obtained by Vortman[4] in 1891 with a stationary anode shows that he always obtained slightly low results in the determination of zinc, mercury, cadmium and antimony in a mercury cathode. His results with lead and bismuth are irregular, but mostly low.

Price also states that upon washing pure mercury with alcohol and ether, a black scum was formed and the mercury lost weight. This phenomenon had been noticed by one of us in some work several years ago, but was ascribed to impurities in either the mercury, alcohol, or ether. If pure mercury washed with pure alcohol and ether lost weight, the method of handling the mercury cathode must be revised. To test this point, some mercury was purified by washing with nitric acid and water, and a supply of pure alcohol and ether prepared. Merck's 99.8 per cent. alcohol was allowed to stand twenty-four hours over freshly prepared quicklime, and then distilled with special precautions over more lime. Ether (labelled "distilled over sodium") was allowed to stand twenty-four hours over small,

[1] Chem. News (1880), **42**, 291, Am. Chem. J., **13**, 571.
[2] Trans. Faraday Soc., **3**, 88, Chem. News, **97**, 99.
[3] Kollock and Smith, J. Am. Chem. Soc., **27**, 1258 and **29**, 797.
[4] Berichte, **24**, 2749.

freshly cut pieces of sodium, and distilled over more sodium with precautions to prevent the absorption of moisture. With the use of these pure materials, neither was there any scum formed on the mercury nor could any mercury be found in portions of the solvents which had stood over night in contact with mercury, and had been decanted therefrom without special precautions.

It seemed possible, then, that Dr. Price's low results were due to impurities in his alcohol or ether; probably the latter, as experiment showed that the black scum was at once formed on washing the purified mercury with ordinary ether from the laboratory bottle.* A standard solution of zinc sulphate was therefore prepared and electrolysed in the apparatus for rapid electroanalysis without rotating electrodes recently described by one of us.[5] The mercury, ether and alcohol were purified as described, and all precautions prescribed by Kollock and Smith[6] were observed. As in the experiments of Kollock and Smith, the dessicator was not evacuated, although Price showed that a decrease in weight, due to loss of ether, was produced by evacuation. Experiments showed that the weight of the cell remained approximately constant, at least for a day or so, and as the mercury would hold the same amount of ether before electrolysis as afterwards, the evacuation was considered unnecessary. The changes in weight found on allowing cells containing zinc or copper amalgam to stand in the dessicator were very slight, being for one day + 0.0001, two days + 0.0003, three days — 0.0003, and — 0.0004, ten days — 0.0005 gram.

To determine whether low results could be due to (1) evaporation of the mercury due to the large amounts of gas liberated on its surface at the high temperature, or (2) solution of the glass by the acid employed, a series of blank determinations was made. The electrolyte consisted of 60-75 c.c. of distilled water acidulated with sulphuric acid. The current ranged from 0.6 — 1.4 amperes, the voltage from 8-16, and the time from 20 to 60 minutes. The changes in weight found were: — 0.0003, ± 0.0000 ± 0.0000, — 0.0001, — 0.0004, — 0.0003, — 0.0001

* It seems quite probable that this scum is caused by hydrogen peroxide, which can often be detected in ether.

[5] Frary, J. Am. Chem. Soc., **29**, 1592; Z. Angew. Chem., **20**, 1897.

[6] J. Am. Chem. Soc., **27**, 1256.

and — 0.0001 gram. The error from these sources appears to be negligible for general work.

The strength of the standard solution of zinc sulphate was determined (a) by evaporation of a measured volume (25 c.c.) in a platinum dish and ignition to constant weight, and (b) by titration with a carefully standardized solution of potassium ferrocyanide. The zinc oxide obtained by method (a) was tested for sulphate, but none was found. The same pipette was used throughout the work, and every precaution taken to insure accurate measurement. The results of the standardization were, method (a) zinc found 0.1367, 0.1369 gram; method (b), 0.1371, and 0.1373 gram; average, 0.1370 gram.

A series of electro-analyses of this solution with the mercury cathode under varying conditions gave the results shown in Table I. The acid used was sulphuric (sp. gr. 1.84).

TABLE I.

Weight of zinc taken in all cases, 0.1370 gram.

No.	Amp.	Volt	Acid (Drops)	Time (Min.)	Zinc left in Electrolyte	Zinc found (Gr.)	Error (Mg.)
1	1.2	10	20	25	. . .	0.1340	—3.0
2	1.2	9	20	30	Trace	0.1320	—5.0
3	1.3	10	25	20	. . .	0.1291	—7.9
4	1.4	11	20	30	None	0.1380	+1.0
5	1.6	13	10	30	"	0.1310	—6.0
6	2.4	12	10	20	"	0.1334	—3.6
7	2.6	20	8	15	. . .	0.1299	—7.1
8	2.9	17	10	15	None	0.1324	—4.6
9	3.0	17	10	15	"	0.1332	—3.8
10	3.0	15	10	15	"	0.1331	—3.9
11	3.0	30	5	20	"	0.1320	—5.0
12	3.1	15	10	15	Trace	0.1321	—4.9
13	3.6	18	10	15	"	0.1297	—7.3
14	4 2	22	10	15	None	0.1256	—11.4

The test for zinc left in solution after electrolysis was made upon the whole volume, by adding potassium ferrocyanide and allowing the test to stand 5 to 10 minutes. Tests with known amounts of zinc sulphate showed that 0.2 mg. of zinc in 200 c.c. was readily detected under these conditions.

In spite of the fact that in most cases the precipitation was

evidently complete, the results, with one exception, are all too low.

During the washing with alcohol and ether it was frequently noticed that fine white particles resembling zinc hydroxide floated in the washings. To ascertain the effect of washing on the amalgam, a weighed cell containing 0.13 gram zinc and 35 grams of mercury was repeatedly washed with water, alcohol and ether, and weighed as in a determination. The weights recorded were 95.6971, 95.6943, 95.6852 and 95.6808 grams. The results account satisfactorily for the errors shown in Table I. The average loss in washing the amalgam was 4.1 mg. per determination, the average error in the analyses in Table I (omitting No. 14) is — 4.7 mg. The washings from the above experiment were separately acidified with nitric acid and concentrated to 20-30 c.c. Upon testing with hydrogen sulphide, a black precipitate (mercuric sulphide) was produced in each case. These precipitates were filtered off, the filtrates combined, concentrated and tested with potassium ferrocyanide. A white precipitate appeared at once, thus showing that the amalgam loses both zinc and mercury during the washing, even with pure alcohol and ether, which do not cause loss on washing pure mercury.

To determine whether all amalgams would act in a similar manner, a series of analyses of a standard copper sulphate solution was made. Four determinations from a sulphuric acid solution using a platinum cathode, gave 0.2169, 0.2170, 0.2172 and 0.2173, or an average of 0.2171 gram copper in 25 c.c.

The determinations made with a mercury cathode were much more satisfactory than those in Table I. The copper amalgam was brighter and cleaner, and no solid particles could be seen in the washings. The presence of free acid seems to have a greater retarding effect with the large volume (60-75 c.c.) of solution which we used than with a small volume,[7] as when only 40 drops of sulphuric acid (sp. gr. 1.84) were added, the precipitation was incomplete after forty minutes. According to the figures given by Kollock and Smith, we should have been able to add 7 c.c. of acid without producing a concentration which would retard precipitation. This was tried on two determinations, with

[7] Kollock and Smith, J. Am. Chem. Soc., **29**, 797.

the result that after 40 minutes a considerate amount (12 and 17 mg.) remained in solution. The results in Table II show that with less acid complete precipitation is readily obtained, and the results are quite accurate. Hydrogen sulphide was used to detect the presence of copper in the solution after electrolysis.

TABLE II.

Copper taken in each case, 0.2171 gram.

No.	Amp.	Volts	Acid (Drops)	Time (Min.)	Copper left in Electrolyte	Copper found (Gm.)	Error (Mg.)
1	1.3	10	20	40	None	0.2167	—0.4
2	1.0	10	20	35	Trace	0.2169	—0.2
3	1.8	13	20	20	None	0.2168	—0.3
4	1.0	8	35	45	"	0.2168	—0.3
5	1.0	10	20	45	"	0.2169	—0.2
6	3.0	20	15	35	Trace	0.2165	—0.6

We are unable to account for the slightly lower results obtained by this method. Upon evaporation of the washings from a determination with nitric acid and testing with hydrogen sulphide, no copper or mercury was found. The liquid remaining from one determination was tested colorimetrically for iron; the amount found was less than 0.1 mg.

Summary.

1. The electrolytic determination of zinc in a mercury cathode gives low results, due to loss of both zinc and mercury in the process of washing with alcohol and ether.

2. Pure mercury does not lose weight appreciably when washed with water, alcohol and ether, nor by liberation of hydrogen on its surface even at high temperatures (100°).

3. The loss in weight during washing seems to be peculiar to zinc, since copper amalgam shows no signs of it, and the determination of copper by means of mercury cathode is quite satisfactory.

University of Minnesota.

October 1, 1908.

DISCUSSION.

DR. H. E. PATTEN: I have read the paper with a great deal of interest, and would like to make the suggestion that in the final writing the authors include the size of the mercury electrode used, that is, its area. You will notice, too, that they state that a certain voltage is used and a certain current. Now, it goes without saying, that to say a certain voltage is used in electrochemical processes means nothing unless the electrode voltage or the resistance of the electrolyte is given. The statement of current intensity conveys no definite meaning, unless we have a statement of the exact current density used.

MR. WALTER T. TAGGERT: In reading over the paper I notice first that attention is called to Wolcott Gibbs as being the originator of the method, which he certainly was, but the application of the method of deposition of metals in mercury, or the quantitative determination of metals, was effected by Prof. Smith, of the University of Pennsylvania.

I next notice in the paper a reference to a method used by Vortman, and it says that Vortman in 1891 noticed a loss in mercury. There is no comparison between the Vortman method and the mercury cup method. Vortman's method is this: He adds to his solution of a zinc salt a weighed quantity of the mercuric salt, and he forms his amalgam on a platinum dish.

PROF. J. W. RICHARDS: I would like to remark that in connection with the statement on page 55, where the writers say, "We are unable to account for the slightly lower results obtained by this method" in the case of copper, and on page 52, where the statement is made that "a series of blank determinations was made" which showed the changes in weight as follows, from 0.003 to 0.004 of a gram. The statement is then made: "The error from these sources appears to be negligible for general work."

I call attention to the fact that the error on page 55, which they say they are unable to account for, is apparently the error of the blank determinations. It varies from .2 up to 6. of a milligram, and the blank determinations are of just about the same order, so I take it that the losses in the blank determinations possibly explain the slight loss in the determination of copper.

MR. FRARY *(communicated)*: The area of the mercury cathode used by us is difficult to estimate, since it consisted of a ring of mercury; the outer diameter of the ring was about 42 mm., and its width about 7 mm. Allowing for the area on the slope of the meniscus at each side, the total area was probably 9-11 cm.^{2}. The only importance attaching to the voltage measurements is that they indicate the approximate amount of heat developed in the cell, which is proportional to the watt-seconds used, and in connection with the heat capacity and rate of radiation would determine the rise in temperature. Since no separation of metals was attempted, and the aim of the research was simply to find out why the method gave low results, I see no reason for determining the cathode potential, especially since it is impractical to regulate it in general electroanalytical work. I should feel inclined to reverse Mr. Patten's statement in regard to current intensity and current density. The amount of metal (plus hydrogen) deposited is a function of the coulombs used, while in the case of a mercury cathode, where we have no trouble with spongy precipitates and non-adherence of the precipitate to the cathode, the current density is of importance only as it may have an effect on the electrode potential. What we require is complete precipitation of the zinc, in the minimum time; we care very little how much hydrogen is precipitated with it.

Mr. Taggert seems to feel that the work of Prof. Smith on the mercury cathode has been slighted; such was not our intention, as is easily seen from our references to it (foot-notes 3, 6 and 7). As to Vortman's method, he precipitated metals as amalgams and obtained low results. An amalgam is an amalgam whether precipitated on a platinum dish or in a glass tube. Our object was to study the behavior of certain amalgams in electroanalysis, so we thought it important to name all who seemed to have obtained poor results by their use.

The description of the apparatus and methods used at Pennsylvania is essentially the same as that given in the paper to which we referred (foot-note 6) except the statement regarding cleansing the apparatus with nitric and chromic acids, which appears to be new. It does not appear what connection there may be between this and the peculiar behavior of the zinc amalgam during the washing with alcohol and ether. Our apparatus had been

cleansed with nitric acid, and was handled with the utmost care.

We stated that we agreed with Kollock and Smith that the retention of ether by the amalgam was negligible.

We considered that prolonged agitation with air and nitric acid was better than distillation of the mercury, as the former method removes the alkali metals which would not be completely removed by distillation. We considered such metals to be the most undesirable impurities, as they, by virtue of their affinity for oxygen, would have the strongest tendency to produce a scum on the mercury.

We hoped to find the method accurate, but were disappointed. If the method does not give good results in the hands of its friends and under the most favorable circumstances, it can hardly be recommended for general work.

Prof. Richards' explanation of the low results in the determination of copper is probably correct, although another cause may be the well-known tendency of copper precipitated on platinum to be contaminated with sulphide, from reduction of the sulphuric acid, and thus our standardizations may have been slightly high.

A paper read in abstract by Prof. W. T. Taggart, at the Fourteenth General Meeting of the American Electrochemical Society, New York City, October 30, 1908; President E. G. Acheson in the chair.

THE DETERMINATION OF ZINC WITH THE AID OF THE MERCURY CATHODE AND ROTATING ANODE.

By Lily G. Kollock and Edgar F. Smith.

It would seem from published statements* that the determination of zinc, when using a mercury cathode and rotating anode, has in the hands of some experimenters given low results. The cause has been variously attributed to the loss of mercury mechanically in washing or chemically by its solution in the acid electrolyte, or that zinc hydrate has been formed and carried away in the wash water. Again it has been said that constant weights could not be obtained unless the amalgam was dried in a vacuum.

This method of determining zinc has been performed hundreds of times in this laboratory by experienced and inexperienced persons with uniform accuracy and the non-occurrence of the discrepancies mentioned by critics of the procedure; so that it seemed proper that we should set forth again our modus operandi as it may be that we failed to record, in previous communications, points in manipulation which were considered perfectly obvious, but which should have been mentioned.

To help those not familiar with the details of the method, we shall endeavor to fully describe the various operations, repeating instructions given in our earlier work and emphasizing precautions which may appear self-evident to one who has worked the method for some time.

It is essential that the cup in which the decomposition occurs should be *clean*. After use and after removal of the mercury the cup should be washed with nitric acid to thoroughly cleanse it from the amalgam which may adhere to the platinum wire or to the glass in the vicinity of the wire. It is next rinsed

* Trans. Faraday Soc., 3, 88, Chem. News, 97, 99.

with water and then washed with chromic acid, followed by water, alcohol and ether. Wipe the outside of the cup, but do not touch the inside with the towel or with the fingers. The cup may be dried in a copper oven, or the ether may simply be allowed to evaporate, and the cup then placed in a desiccator ready to be used. Keep 100 c.c. of pure mercury, previously washed with absolute alcohol and ether, in a desiccator, to be drawn upon as occasion may demand. Fifty to sixty grams are introduced into each cup, and this portion will serve for a number of determinations. Remove with a camel's hair brush or a feather any minute globules of mercury adhering to the outside of the cup or around the rim. Cover the cup containing the mercury, after weighing, with a small watch crystal. Never move or carry the cup without this cover. After introducing the electrolyte by means of a pipette, and making proper dilution, place the cup on a copper plate connected with the cathode, and raise the whole to a proper position with respect to the anode. Place the cover glasses over the cup, cause the anode to rotate and let the current pass through the electrolyte. Toward the end of the decomposition wash the cover glasses and sides of the tube with a stream of water from a wash bottle. The condensation of the steam, when the current is high, if sufficient to remove all of the electrolyte which may have been carried to the sides or the top of the cup by rotation or by the escaping gases.

When the decomposition is completed, stop the motor, remove the cover glasses, pour distilled water into the cup from a wash bottle, and siphon out the liquid until the level almost reaches the spiral portion of the anode. Then refill the cup and repeat the operation until the current falls to zero—that is, until practically all of the acid solution has been removed. As much as 300 c.c. of water are used for the purpose. Now interrupt the current. That the acid liquid must be removed before the current is broken is a precaution which must be observed, otherwise the acid will act upon the amalgam and cause a resolution of the deposited metal. Several experiments were made to prove this. After the zinc had been deposited, the current was broken. The voltmeter was still in connection with the cup. It acted as an electrolytic cell having a pressure of 2.5 volts and zinc dissolved.

The deposit, on weighing, showed a loss of a milligram or more, and the solution, afterward poured from the cup, gave a distinct precipitation with potassium ferrocyanide. The longer the acid acted, the greater was the loss in weight. When water was added and the solution quickly siphoned out, the deposit lost 0.0004 gm., and the wash-water gave a turbidity with potassium ferrocyanide. In a previous paper this precaution was stated explicitly, and Price and Dodge confirm it, because they declare in their first paper that zinc was not found in the siphonate when the current was not interrupted, but it was found there whenever the current was interrupted.

Next remove the cup and pour off the greater part of the remaining liquid by inclining the tube to the horizontal position. Do not permit the amalgam to come to the edge of the cup or in contact with the finger which is held at the top of the cup in order to hold it more firmly. The cup must then be brought back carefully to a vertical position, and pour in it about 10 c.c. of absolute alcohol. Rotate the cup, after it has been inclined to an angle of 45°, in order that the entire mass of mercury may be brought in contact with the alcohol. The greater part of the alcohol is then allowed to flow off in the same way as described above for the removal of the water. Repeat this operation with a second and third portion of alcohol, then with two or three portions of anhydrous ether.

Let the cup, after the last washing with ether, stand for a few minutes until the greater part of the ether has evaporated, then place it in a desiccator. A vacuum desiccator was not found necessary. Any trace of ether remaining may be eliminated by gently twirling (rotating) the cup at an angle of 45°.

For the present work, zinc sulphate was recrystallized several times, carefully dried, and its solution made so that 5 c.c. contained 0.1316 gram of zinc. The results obtained from about 40 experiments show the error to be very slight. The results were recorded as they were obtained. No selection from them was made. In two trials the error is greater than is permissible, but this is explainable by certain conditions prevailing at the time the experiments were performed. The sulphuric acid set free was determined in the greater number of the experiments. This was accomplished by titration with $\frac{N}{5}$ sodium carbonate, using

methyl orange as an indicator. These results are also in accord with the zinc values.

Zinc was not found in the washings on testing with potassium ferrocyanide. To ascertain whether mercury or zinc was lost in the wash water, the siphonate from 12 determinations was evaporated to about 10 cubic centimeters, a little nitric acid was added, and the solution evaporated to a very small volume, then diluted to about 10 c.c. A bright piece of copper foil was introduced into this solution. No evidence of mercury could be detected. Zinc was also absent. This test was repeated with many other determinations. The washings with alcohol and ether were evaporated and treated in the same way. No mercury or zinc could be discovered.

Zinc hydrate was never discovered in any of the trials. But to find out what would take place under other than our own conditions, it was noticed if water remained in contact with the amalgam for some time that there was a separation of zinc hydroxide. In our own experiments, alcohol was always added quickly to the amalgam. It is necessary, therefore, to be expeditious at this point. For this reason, as well as for rapid drying, use absolute alcohol and ether.

The following are the conditions under which our experiments were conducted, and the results are appended:

Volume of solution = 10 c.c.

Current in amperes, ⅓.

E. M. F., 8.5 - 5 volts.

Rotation of anode, 550 - 750 revolutions per minute.

Time, 12 - 15 minutes.

Cathode surface, 20 sq. cm.

Experiment	Zinc Found in gram	Error Zinc	H_2SO_4 In gram	H_2SO_4 Error in gram
1	0.1319	+.0003	.1985	+.0001
2	.1317	+.0001	.1987	+.0003
3	.1316	. . .	.1986	+.0002
4	.1314	—.0001	.19845	+.00005
5	.1318	+.0002	.1984	. . .
6	.1317	+.0001	.1987	+.0003
7	.1316	. . .	.1983	—.0001
8	.1313	—.0003	.1988	+.0004
9	.1315	—.0001	.1986	+.0002
10	.1319	+.0003	.1989	+.0003
11	.1317	+.0001	.1989	+.0005
12	.1317	+.0001	.1987	+.0003
13	.1310	—.0006	.1984	.
14	.1313	—.0003	.1985	+.0001
15	.1316	. . .	.1986	+.0002
16	.1313	—.0003	.1985	+.0001
17	.1316	. .	.1987	+.0003
18	.1312	—.0004	.1980	—.0004
19	.1314	—.0002	.1984	. . .
20	.1315	—.0001	.1987	+ 0003
21	.1317	+.0001	.1980	—.0004
22	.1319	+.0003	.1984	. . .
23	.1315	—.0001	. . .	. . .
24	.1316	. . .	. . .	. . .
25	.1318	+.0002	. . .	. . .
26	.1310	—.0006	. . .	. . .
27	.1314	—.0002	. . .	. . .
28	.1318	+.0002	. . .	. . .
29	.1317	+.0001	. . .	. . .
30	.1316	. . .	. . .	. . .
31	.1312	—.0003	. . .	. . .
32	.1318	+.0002	. . .	. . .
33	.1316	. . .	. . .	. . .
34	.1313	—.0003	. . .	. . .
35	.2630	—.0002	.3970	+.0002
36	.1314	. . .	. . .	. . .
37	.1314	—.0002	.1987	+.0003
38	.1318	+.0002	.1981	—.0003
39	.1317	+.0001	.1987	+.0003

It is, therefore, evident, despite the difficulties of some of our friends, that this method for the determination of zinc is reliable, accurate and rapid.

Reference to our former publications on this subject will also reveal that many other metals are just as readily and accurately estimated by means of the mercury cathode and rotating anode.

University of Pennsylvania.

DISCUSSION.

CARL HERING *(communicated):* In connection with the deposition of zinc on to a mercury cathode, the following curious observation made by the writer some years ago may perhaps be of some interest, although it has no direct bearing on electro-analysis; it refers more directly to the efficiency of the process, and this is of no importance in analytical work.

The anode was a flat horizontal disc, placed about a half inch above the mercury in the bottom of the beaker, and it was smaller in diameter than the exposed surface of the mercury. The solution was a strong solution of zinc sulphate, quite strongly acidulated with sulphuric acid; it was the product of an exhausted battery. With low current densities there was the usual gassing all over the cathode, as the sulphuric acid is more easily decomposed than the zinc sulphate. But on raising the current density to a high value the gassing ceased completely immediately under the anode and over an area about equal to that of the anode, while outside of this area the gassing was very profuse. The line of demarcation between them was sharp. The non-guessing surface of the mercury appeared agitated, indicating that something was going on there, unless it was due to the riplets produced by the gassing area. The major part of the current was presumably passing through this non-gassing area. On moving the anode, this non-gassing area followed all such motions. As it is generally understood that it is impossible to decompose zinc sulphate without also decomposing some of the more easily decomposed acid whereby hydrogen is set free, the result is of interest, as it appears to contradict this. The voltage was of course much higher than that theoretically required for reducing the zinc, hence the loss in energy was probably greater than that for decomposing the acid at lower current densities. The process may therefore have no value commercially, for which reason no further researches were made at that time. Moreover, the solution becomes quite warm, showing a very poor efficiency.

Another observation was that when such zinc amalgam was thoroughly washed in water a heavy, bluish-gray powder separated out in relatively large quantities. It looked like pulverized zinc. The original solution in this case also contained some chromium salts, as it was the spent liquid from a zinc-bichromate battery. It was thought at the time that this powder may have been chromium.

Paper read at the Fourteenth General Meeting of the American Electrochemical Society, New York City, October 31, 1908; President E. G. Acheson in the Chair.

EQUILIBRIA IN STANDARD CELLS.

By G. A. Hulett.

The two combinations which have shown the greatest reproducibility and constancy are the Clark cell and the Weston or Cadmium cell, and these are very similar in construction:

Zinc amalgam / $ZnSO_47H_2O$, Hg_2SO_4, H_2O / mercury

Cadmium amalgam / $CdSO_48/3\ H_2O$, Hg_2SO_4, H_2O / mercury

Many other combinations have been suggested and tried, but have generally been found wanting in constancy or reproducibility. It has been the universal experience that a metal in the solid state gives an inconstant potential against a solution even of its own salts, and, while the cause of this is not fully understood, the fact has restricted the choice of materials to liquid electrodes for standard cells. Thus mercury forms one electrode and a liquid amalgam the other. For this amalgam it is desirable that the base metal be well towards the positive end of the electrochemical series and thus far removed from mercury, and that it have a fair solubility in mercury. Gouy (J. de Physiq. 4,320) gives as the solubility of metals in mercury: Cadmium 4 per cent., zinc 1.8 per cent., lead 1.3d per cent., bismuth 1.2 per cent., tin .6 per cent., gold .13 per cent., and distinctly less for other metals. It is further desirable that the base metal exist in only one state of oxidation and that its salts be well defined. In these requirements zinc and cadmium are distinctly superior to all other metals, and it is improbable that the other metals will be found to be of service in standard cells. Furthermore, the depolarizer must have a certain solubility, since mercury shows a constant potential only against electrolytes which contain definite amounts of mercury in solution, and experience has shown that this solubility must not be too small, as in the case of the calomel cell, or too great, as in the case of the Daniell cell, where the depolarizer, copper sulphate, is quite soluble. Since mercury is the

cathode, the depolarizer must be a mercur*ous* salt with a solubility of a few parts in a thousand, and there are very few available salts of mercury answering to these requirements; mercurous sulphate is distinctly the best, although mercurous chlorate deserves to be considered. It is also to be noted that the acid radical of the base metal must be the same as that of the depolarizer, and if mercurous sulphate is used as depolarizer, sulphate of zinc or cadmium must be used in the construction of the cell. There are then excellent reasons for devoting attention to the Clark and cadmium cells rather than seeking for other combinations.

The Cathode Systems.—The potential difference of an amalgam against an electrolyte varies with the concentration of the metal in the amalgam, and so this concentration must be fixed and reproducible for any given temperature. This requirement is attained by utilizing a saturated solution of the metal with an excess of the solid, so that at each temperature the proper concentration is automatically produced whether the electrode has lost or gained metal from the electrolyte or in other ways. In the Clark cell this two-phase system is made up of zinc and a saturated amalgam, while in the cadmium cell the solid phase in the amalgam is an isomorphous mixture of mercury and cadmium which has a definite composition for each temperature as well as the liquid amalgam in equilibrium with it. The liquid phase of this cadmium amalgam at 25° has been carefully analyzed and found to contain 5.903 g. of cadmium to 100 g. of mercury, and it is probably one of the most accurately reproducible concentrations known. In some experiments on the e. m. f. of cadmium amalgams observations were made on two amalgams containing 10 per cent. and 7 per cent. of cadmium. These formed two phase systems with different amounts of the solid phase, and were used as electrodes in a cadmium sulphate solution. The system was freed of oxygen and hermetically sealed in a glass cell. The observations extended over a period of six months, and, although the system was subjected to wide variations of temperature, whenever it was brought to 25° equilibrium was soon established and there never was an e. m. f. between the amalgams greater than .0000012 volts; it was generally less than this, but if this difference was all due to differences in the concentration of

cadmium in the two liquid phases, it indicated differences of only 7 parts in a million. In order that this two-phase amalgam may give a definite p.d. it is necessary that it be in contact with a solution which contains a cadmium salt of definite concentration, and here again advantage is taken of the property of a saturated solution with an excess of the solid salt. Crystallized cadmium sulphate, $CdSO_4\ H_2O$ is a particularly well defined salt and gives a concentratoin of 3.363 miles in a liter at 25° with a density of 1.6176.

Since the anode is made up of two metals, cadmium and mercury, it is essential for equilibrium that the electrolyte contain in solution salts of *both metals,* and that there be a definite ratio between the concentrations of these salts depending on the relative concentration of the metals in the electrode and upon their relative solution pressures or positions in the electrochemical series. The most thoroughly investigated case of this kind is the equilibrium between mercury, silver and a solution of their nitrates[1]. It was shown by these authors that silver did not completely remove mercury from solution, and that mercury when brought into contact with a silver nitrate solution did go into solution and reduce an equivalent amount of silver. From whichever side they approached there resulted a definite equilibrium with both silver and mercury in the metal phase and in the solution. The equilibrium was represented by the following relation proposed by Nernst (Zeit. Phys. Chem., 22,537)

$$\frac{n_1}{\sqrt{\frac{P_1}{p_1}}} = \frac{n_2}{\sqrt{\frac{P_2}{p_2}}}$$

where P_1 and P_2 represents the solution pressures of the two metals and p_1 and p_2 the osmotic pressures of the ions of these metals in the solution, and n_1 and n_2 represent their respective valencies. It was further pointed out by Reinders that in case the metals formed a solution (liquid or solid) that P_1 and P_2 would vary with their respective concentrations in the metal phase and the equation was correspondingly modified.

It must be concluded, therefore, that cadmium will not completely remove mercury from solution, and that if mercury is brought into contact with a cadmium sulphate solution some of the

[1] Ogg, Z. Phys. Chem., **27**, 285. Reinders, Z. Phys. Chem., **54**, 609.

mercury will go into solution and force out an equivalent amount of cadmium. Reinders[2] suggested that this state of affairs existed in the standard cells, but due to the wide difference between cadmium and mercury in the electrochemical series, the equilibrium concentration of mercury in the anode leg must be very small. Observations on the cells themselves tend to confirm this point, since the anode shows its correct p.d. soon after a cell is constructed, later mercurous sulphate diffuses over from the cathode and then there is a certain concentration of mercury about the amalgam, but the arrival of the dissolved mercury at the anode is not accompanied by a change in the p.d. so it would seem that the equilibrium concentration of mercury about the anode was already present before the mercury diffused over from the cathode, having been furnished by the interaction of the amalgam and the solution. Some results on the e. m. f. of dilute amalgams, which will soon be published, give more direct evidence of this relation and indicate that the mercury concentration is very small, but it is to be remembered that it is not the absolute concentration but the relative ones which are of importance in determining electromotive forces.

The anode of a standard cell may then be regarded as made up of an electrode in two phases; the liquid amalgam has an exceedingly exact and reproducible concentration for each temperature, and when this equilibrium is established the solid phase has of course the same potential as the liquid phase, and does not show the irregularities exhibited by a solid metal electrode. The electrolyte about this electrode is saturated with cadmium sulphate, and an excess of the crystals insures a definite concentration of cadmium in the electrolyte for each temperature, while the interaction of the amalgam and this solution furnishes an equally definite mercury concentration in the liquid surrounding the electrode. The anode system is thus seen to be made up of five phases: Solid and liquid amalgam, solution, solid salt of the base metal, and vapor. The components may be taken to be: Mercury, base metal, water and acid radical, and thus for each temperature they are nonvarient systems. It has been found that the equilibria in these anode systems are rapidly established as soon as the temperature is fixed.

[2] Z. Phys. Chem., **42**, 232.

The two anode systems it is probable that they are equally reproducible and constant; the solubility of zinc sulphate crystals changes very greatly with the temperature and there is a tendency for these crystals to "cake" above the amalgam, while the cadmium sulphate crystals have an exceptionally small temperature-solubility coefficient and have not shown this tendency. The tendency of the zinc amalgam to cause the platinum contact wire to crack the glass has been a source of trouble with Clark cells, also the zinc amalgam slowly generates hydrogen from its sulphate solution and it is probable that a zinc sulphate solution has a fair concentration of hydrogen ions, while there is no evidence of the liberation of hydrogen in the anode system of the cadmium cell and it is probable from other experiments also that a cadmium sulphate solution is only slightly if at all hydrolysed and so contains practically no hydrogen ions. The cadmium anode is more completely reversible than the zinc anode for, in as far as hydrogen is liberated with the deposition of the metal, the change is not reversible, but this does not effect the equilibrium, reproducibility or constancy of the zinc anode system, and while the lack of hydrogen ions in the cadmium sulphate solution is an advantage in the anode system of the cadmium cell, it is the cause of a state of unstable equilibrium in the cathode system of this cell as the following results show. In this respect the acid qualities of the zinc sulphate solution constitute a very decided advantage.

Renders[3] was the first to point out that these cells could not be considered as one system since the electrolyte at the cathode is saturated with mercurous sulphate which contniually diffuses over to the anode where it is reduced to a very small concentration by the amalgam, and equilibrium would be established only on the disappearance of the base metal from the anode or the disappearance of the mercurous sulphate as a solid phase; in either case the e. m. f. would become zero or inconstant. This type of cell is made up of two distinct sysetms which are in electrolytic contact but not in equilibrium with each other, but as the cells are constructed the diffusion from cathode to anode is so slow that the life of the combination is very great. It was further noted by Reinders that, for a constant and reproducible e. m. f.

[3] Konink, Akad. Wetensch., Amsterdam, Versl. **11**, 115.

it was essential that both the anode system and the cathode system be nonvarient systems when the temperature was fixed. The Clark cell conforms to these requirements, but the results now at hand show that the cathode system of the cadmium cell does not.

The Cathode Systems.—The materials used in constructing the cathode system of the cell are: Mercury, mercurous sulphate, cadmium sulphate and water. An excess of the two sulphates are used and it may be assumed that there results a solution saturated in respect to $CdSO_4$ H_2O and Hg_2SO_4, provided that these salts are in their stable forms at the temperatures considered, do not form isomorphous mixtures double salts or interact chemically. The mercury will not be pure but will contain a small equilibrium concentration of cadmium due to its interaction with the solution. There appears then a p.d. between this electrode and the solution which is a function only of the temperature, and if the premises are correct it may be concluded that rotating this system will not affect the p.d. provided the materials are not reduced to such a fine state of subdivision that the effect due to surface tension becomes measurable. But it has been found[4] that there was a very decided change in the p.d. when this cathode system of the cadmium cell was rotated; a slow increase of potential was observed amounting in some cases to two millivolts. The apparatus used in rotating the system gave a very thorough mixing of the contents but avoided grinding the solids to a fine powder or reducing the mercury to small globules; indirect evidence was also obtained to show that the observed change in p.d. was in no way due to surface tension. It seemed possible that the dissolved oxygen had changed some of the mercurous sulphate to the more soluble mercuric form and that the resulting increased mercury concentration had caused the increased p.d. This obvious explanation was rigidly tested[5] but it was found that the absence of oxygen made no difference in the results of the rotation experiment, and considerable indirect evidence pointed to this same conclusion. The evidence on this point seems so decisive that it is to be concluded that dissolved oxygen plays no role whatever in these systems. If the *rate* of solution of mercurous sulphate was slow the equilibrium point would be only

[4] Phys. Rev., **23**, 176 and **25**, 16.

[5] Phys. Rev., **25**, 19-24.

slowly approached and only on rotating the system, but it has been frequently noticed that the cell assumes a certain e. m. f. soon after it is constructed and if its value undergoes any change it is to decrease, while an increasing mercury concentration would means an increasing e. m. f. This fact seems to effectively dispose of the idea of a slow rate of solution of the mercurous sulphate in a cadmium sulphate solution; it shows that, at least the layer next to the mercury electrode, dissolved as much mercury sulphate as it could in a very short time.

There was still the possibility that the mercurous sulphate was hydrolysed by the cadmium sulphate solution with the formation of a basic salt and an acid in solution, a reaction which would proceed until a certain acid concentration was established. This point was tested by adding acid to the system and it was found that it did not then show the slowly increasing p.d. but came to a constant value very soon after the rotation was begun. This was good evidence that mercurous sulphate, conforming to its well known tendency, did hydrolyse in a cadmium sulphate solution.

The only evidence for the increase in the mercury concentration was the increase in the p.d. observed in the rotation experiments; this increase of some two millivolts would indicate an increase of some 15 per cent. in the concentratoin of the mercurous sulphate, acording to Nernst logarithmic relation, assuming that the gram of mercurous sulphate in a liter of the solution was largely dissociated, but if it was only slightly dissociated in the cadmium sulphate solution the observed change in p.d. would indicate a much greater increase in the mercury concentratoin and it seemed possible to analytically detect such changes in concentration if they existed.

The clear solutoin from the cathode system had a density of 1.6176, contained 760 g. of $CdSO_4$ in a liter, but only about one gram of mercurous sulphate, and considerable difficulty was encountered until a special method of analysis was devised. It was found that an excess of hydrochloric acid entirely prevented the precipitation of CdS by sulphuretted hydrogen, but all the mercury was thrown out as a black sulphide, as this is one of the most insoluble substances known. The difficulty of separating this sulphide of mercury from the viscous liquid was so great,

however, that it could not be used as a quantitative method, although it served as a method of detecting traces of mercury in the concentrated cadmium sulphate solution and also to estimate small quantities colorimetrically.

Hydrochloric acid was found to precipitate mercury from these solutions as mercurous chloride which Ley and Himbuchsen[6] have shown to have a solubility of only .2 mg. in a liter, much less soluble than silver chloride. It was found that the mercurous chloride soon coagulated and filtered nicely. It is known, however, that the solutions also contain a certain very small concentration of mercur*ic* mercury, since there is always an equilibrium established between the mercurous and mercuric mercury in a solution which is in contact with mercury, but in the case of mercury this equilibrium favors the *-ous* state of oxidation so that probably less than one per cent. of the mercury in solution was in the *-ic* state. In consideration of this point it was deemed best to follow the precipitating reagent, HCl, with a reduction agent, and sulphur dioxide was used. These reagents so completely removed the mercury that sulphuretted hydrogen failed to show a trace of mercury in the filtrate. The mercurous chloride was easily collected in a Gooch crucible, washed with water and dried in a vacuum desiccator over calcium chloride[7]. It had been previously shown that this precipitate could be completely dried in this way without loss while it is noticeably volatile at 100°, and again it has been found that this is a most satisfactory analysis and an exceedingly exact method of determining mercury.

As soon as it was found possible to determine the mercury quantitatively in these cathode solutions the rotation experiments were repeated and a complete analysis was made of the clear solutions together with the density, conductivity and p.d. against mercury. These determinations have been made also on systems to which known amounts of sulphuric acid were added, and from these results the solubility curve of mercurous sulphate as a function of the sulphuric acid concentration in this system has been determined and the curve has given some definite information

[6] Z. f. Electrochem., **10**, 301.

[7] Z. Phys. Chem., **49**, 500.

on the hydrolysis of mercurous sulphate in a cadmium sulphate solution.

Materials, Apparatus and Method.—In the previous experiments[8] it was found that either the electrolytic mercurous sulphate or the chemically prepared salt gave high values in the rotated cathode system of the cadmium cell. The essentials are that the depolarizer be normal mercurous sulphate, that acid be rigidly excluded from the system and that the system be closed during the rotation. For the present work the mercurous sulphate was prepared by adding, drop by drop, a molecular solution of mercurous nitrate to a rapidly stirred sulphuric acid solution. Two liters of this acid, density 1.15, were used in preparing 125 grams of the mercurous sulphate. The stirring was done with a motor and three hours were taken for the precipitation so as to obtain a well crystallized product free of inclusions. The action of light was avoided, while, with the excess of acid used and metallic mercury in both solutions, the basic salts could not have formed. The product was pure white, distinctly crystalline and easily washed, but as the complete removal of acid was important, particular attention was given to this operation. The salt was first thoroughly washed with molecular sulphuric acid, then with absolute alcohol, and finally with some of the solution in which it was to be used. This was done in a Gooch crucible with filter disk and strong suction after each washing. Cells have been constructed from time to time with this preparation and they have shown the normal value and have remained constant. The cadmium sulphate was recrystallized and only the clear, well-rinsed crystals used, as this salt is not isomorphous with other sulphates, impurities were not feared. The zinc sulphate was more troublesome to obtain in a satisfactory state; iron, lead and cadmium were removed by the method of Mylius and Fromm[9]. The salt was then recrystallized and the crystals centrifuged (Richards). The mercury used was chemically purified with an acid solution of mercurous nitrate and then distilled under diminished pressure with a current of air passing through with the vapor[10], a method that insures the removal of such metals as zinc and cadmium.

[8] Phys. Rev., **23**, 177, and **25**, 19.
[9] Z. Anorg. Chem., **9**, 144.
[10] Phys. Rev., **21**, 388.

Conductivity water was used where justified in making the solutions, and where acid was to be added to the system the zinc or cadmium sulphates were dissolved in sulphuric acid of known strength. These acids were all made by dilution from the original "molecular sulphuric acid" which was made from distilled sulphuric acid and conductivity water; the density of this molecular sulphuric acid solution was 1.05948 at 25° and it was found to be 1.012 molar by comparison with a carefully analyzed hydrochloric acid solution.

FIG. 1.

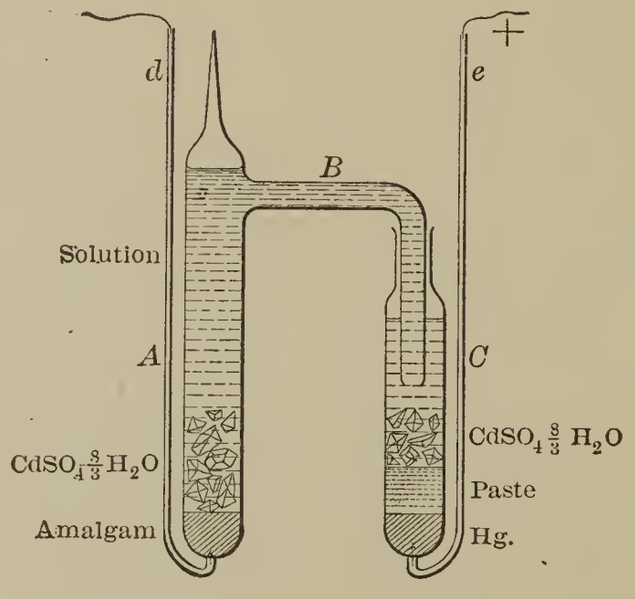

FIG. 2.

The systems were brought to equilibrium in tubes 32 mm. in diameter and 150 mm. long, made as shown in Fig. 1. A tube was charged with about 100 g. of mercury, a good excess, then with about 20 g. of the crushed zinc or cadmium sulphate crystals and 25 g. of the properly washed mercurous sulphate crystals; finally the tube was filled to the neck with the saturated zinc or cadmium sulphate solution. Attention was given to really saturating these solutions at about 25°. The tube was now closed with a cork, and a rubber cap was drawn over the cork and neck of the tube to securely close the system. The tube was rotated in the bath in a horizontal position and about the longer axis. It rested in the loops of little belts which extended down from a

shaft which was fixed above the bath and its speed so regulated that the tube rotated at a rate of 10 revolutions a minute. This rate gave a most thorough mixing of the contents of the tube but was not rapid enough to grind the solid contents to a powder or to cause the mercury to become divided into fine globules. From time to time the tube was brought to an upright position in the bath, the cap and cork were removed, and the anode A of the adjustable cell, Fig. 2, was brought into contact with the contents of the tube and thus the e. m. f. between the amalgam of this anode and the mercury of the system was determined. Whatever changes in the e. m. f. were observed between successive observations were known to be due to changes in the p.d. of the rotated cathode system because the anode was not rotated, and with its own unrotated cathode C (Fig. 2) there was always a check on the constancy of the anode and also a comparison of the rotated and unrotated system since the same materials served for the construction of both of these systems. Each of the equilibrium tubes was rotated until the potential difference of the system became constant, and in some cases this required two weeks or more. It is to be noted that during the rotation nothing could enter or leave the system nor at any time since the system was quiet when the anode was adjusted and a diffusion of dissolved mercury out through the narrow tube B of the anode tester during the time of making a measurement was not to be considered[11].

When the system was in equilibrium, as indicated by no further change in the e. m. f., the tube was allowed to stand quietly in the bath for a day, or until the liquid was perfectly clear, and then the solution was drawn up into a large pyknometer through a little glass tube. The pyknometer and contents were brought to 25° and thus the mass of a known volume of the solution was determined. This solution was transferred, with a little water, to a flask, and made acid with sulphuric acid and then the mercury was precipitated with 5 ccm. of strong hydrochloric acid, and

[11] Attention is called to this point since it will be readily seen that if the anode and cathode systems had been in liquid contact during the rotation that the diffusion of mercurous sulphate to the anode, aided by convection, would have been a maximum, and since the hydrolysis of mercurous sulphate in this system is an exceptionally slow reaction, there would have been little change in the mercury concentration in the cathode system, such methods would not show the real changes that take place. The neglect to guard against this may possibly account for the low results obtained in some of the experiments lately made by Drs. F. A. Wolff and C. E. Waters. Bull. Bureau of Standards, Vol. 4, 81.

finally sulphur dioxide was bubbled into the solution to reduce the small amount of mercuric mercury. The HgCl coagulated nicely and required only a very thin felt in the Gooch crucible; it was determined as already described (p. 9) and the weight calculated to Hg_2SO_4. The results are regarded as accurate to .1 mg. The clear filtrate was evaporated in a platinum dish, heated to expel the hydrochloric acid and excess of sulphuric acid and, after cooling in a desiccator, weighed. The dish and sulphate was then reheated for an hour at about 500° in an atmosphere containing a little SO_3 vapor, cooled and re-weighed.

Thus for each system the mass of the solution, of the $CdSO_4$, and the mercury as HgCl, were directly determined while the water was had by difference. In case acid had been added a further calculation was necessary, for example, Experiment No. 1, "Cadmium system in molar sulphuric acid"—the dissolved $CdSO_4$ 8/3 H_2O added its water of crystallizatoin to the solution, but the amount was obtained from the weight of the $CdSO_4$ and with this the amount of H_2SO_4 in the volume of the solution taken was determinable, in this experiment where molar sulphuric acid had been used the solution was only .810 molar in respect to sulphuric acid.

The thermostat used in the previous work[12] maintained the temperature to within .02° of 25° throughout long runs and materially aided in the accuracy of many of the measurements. The temperature was determined by a 1/50° calibrated thermometer which was checked at 32.383°, the transition point of sodium sulphate (Richards and Wells). The two pyknometers used were calibrated from time to time and it was found that the mass of the pyknometer, alone or filled with water, was determinable to less than ½ mg. The volumes were: No. 1, 47.7605, and No. 2, 49.0856 ccm³ at 25°. All weighings were made by the substitutoin of calibrated weights and, where justifiable, vacuum corrections were made. The conductivity measurements were made in the usual way; a long narrow "Arrhenius" cell with a capacity factor of 8,040 was used.

In the e. m. f. determinations the same precautions and apparatus were used as in the previous work[13], it was found

[12] Phys. Rev., **25**, 19.

[13] Phys. Rev., **25**, 29.

quite necessary to thoroughly insulate the lead wires to the rotation tubes and for the adjustable cells, since they dipped into the water bath; this was done by covering them with glass tubes as indicated in Figs. 1 and 2.

CATHODE SYSTEMS OF THE CADMIUM CELL.

Experiment 1, system in 1.012 molar sulphuric acid, roated 8 days, 25°.

				Moles in a liter.
Mass of solution in	49.0856 c. cm.	75.377	g.	
" " $CdSO_4$	" " "	27.384	"	2.6757
" " Hg_2SO_4	" " "	.0371	"	.001524
" " H_2O	" " "	44.051	"	48.683
" " H_2SO_4	" " "	3.905	"	.810
D_{25}	1.53564	E. m. f.	1.01678	V.
λ_{25}	.14423	Adjustable cell	1.01671	"

Experiment 2, system in .506 molar sulphuric acid, rotated 5 days, 25°.

				Moles in a liter.
Mass of solution in	47.7605 c. cm.	75.209	g.	
" " $CdSO_4$	" " "	29.989	"	3.0116
" " Hg_2SO_4	" " "	.0442	"	.001867
" " H_2O	" " "	43.330	"	50.860
" " H_2SO_4	" " "	1.847	"	.384
D_{25}	1.57511	E. m. f.	1.01747	V.
λ_{25}	.07738	Adjustable cell	1.01745	"

Experiment 3, system in .2024 molar sulphuric acid, rotated 6 days, 25°.

				Moles in a liter.
Mass of solution in	47.7605 c. cm.	76.433	g.	
" " $CdSO_4$	" " "	32.0905	"	3.2226
" " Hg_2SO_4	" " "	.0478	"	.002017
" " H_2O	" " "	43.570	"	50.641
" " H_2SO_4	" " "	.7263	"	.155
D_{25}	1.60032	E. m. f.	1.01808	V.
λ_{25}	.05350	Adjustable cell	1.01802	"

Experiment 4, system in .1012 molar sulphuric acid, rotated 10 days, 25°.

				Moles in a liter.
Mass of solution in	47.7605 c. cm.	76.836	g.	
" " $CdSO_4$	" " "	32.805	"	3.2944
" " Hg_2SO_4	" " "	.04937	"	.002084
" " H_2O	" " "	43.622	"	50.700
" " H_2SO_4	" " "	.360	"	.0769
D_{25}	1.60880	E. m. f.	1.01823	V.
λ_{25}	.04674	Adjustable cell	1.01821	"

Experiment 5, system in .05064 molar sulphuric acid, rotated 7 days, 25°.

Mass of solution in	47.7605 c. cm.....	77.032 g.	Moles in a liter.
" " $CdSO_4$ "	" "	33.120 "	3.3260
" " Hg_2SO_4 "	" "	.05176 "	.002185
" " H_2O "	" "	43.680 "	50.722
" " H_2SO_4 "	" "	.180 "	.0384
D_{25} 1.61289	E. m. f.....	1.01834 V.	
λ_{25} .... .04372	Adjustable cell.....	1.01834 "	

Experiment 6, system in .02024 molar sulphuric accid, rotated 13 days, 25°.

Mass of solution in	49.0856 c. cm.....	79.304 g.	Moles in a liter.
" " $CdSO_4$ "	" "	34.262 "	3.348
" " Hg_2SO_4 "	" "	.05514 "	.002265
" " H_2O "	" "	44.914 "	50.910
" " H_2SO_4 "	" "	.0737 "	.0153
D_{25} 1.61565	E. m. f.....	1.01847 V.	
λ_{25} .... .04180	Adjustable cell.....	1.01835 "	

Experiment 7, system without acid added, rotated 21 days, 25°.

Mass of solution in	47.7605 c. cm.....	77.251 g.	Moles in a liter.
" " $CdSO_4$ "	" "	33.470 "	3.361
" " Hg_2SO_4 "	" "	.0640 "	.002701
" " H_2O "	" "	43.717 "	50.800
D_{25} 1.61747	E. m. f.....	1.01905 V.	
λ_{25} .... .04037	Adjustable cell.....	1.01837 "	

Experiment 8, system without acid added, rotated 21 days, 25°.

Mass of solution in	49.0856 c. cm.....	79.405 g.	Moles in a liter.
" " $CdSO_4$ "	" "	34.435 "	3.365
" " Hg_2SO_4 "	" "	.0726 "	.002980
" " H_2O "	" "	44.897 "	50.770
D_{25} 1.61771	E. m. f.....	1.01940 V.	
λ_{25} .... .04057	Adjustable cell.....	1.01836 "	

Experiment 9, system without acid added, fresh solution was added to the solids of experiment 8, rotated 20 days, 25°.

Mass of solution in	49.0856 c. cm.....	79.402 g.	Moles in a liter.
" " $CdSO_4$ "	" "	34.417 "	3.363
" " Hg_2SO_4 "	" "	.0742 "	.003047
" " H_2O "	" "	44.911 "	50.790
D_{25} 1.61765	E. m. f.....	1.02015 V.	
λ_{25} .... .04061	Adjustable cell.....	1.01836 "	

CATHODE SYSTEM OF THE CLARK CELL.

Experiment 10, system with 1.012 molar sulphuric acid, rotated 8 days, 25°.

Mass of solution in 49.0856 c. cm.....	72.2942 g.		Moles in a liter.
" " $ZnSO_4$ " " "	24.000 "		3.028
" " Hg_2SO_4 " " "	.0296 "		.001216
" " H_2O " " "	45.500 "		51.460
" " H_2SO_4 " " "	2.765 "		.5744
D_{25} 1.47436	E. m. f.....	1.41588 V.	
λ_{25} .... .08588	Adjustable cell.....	1.41595 "	

Experiment 11, system with .506 molar sulphuric acid, rotated 3 days, 25°.

Mass of solution in 49.0856 c. cm.....	72.7524 g.		Moles in a liter.
" " $ZnSO_4$ " " "	25.355 "		3.1993
" " Hg_2SO_4 " " "	.03516 "		.001444
" " H_2O " " "	46.032 "		52.060
" " H_2SO_4 " " "	1.330 "		.270
D_{25} 1.48214	E. m. f.....	1.41813 V.	
λ_{25} .... .06110	Adjustable cell.....	1.41810 "	

Experiment 12, system with .1012 molar sulphuric acid, rotated 3 days, 25°.

Mass of solution in 49.0856 c. cm.....	73.155 g.		Moles in a liter.
" " $ZnSO_4$ " " "	26.402 "		3.331
" " Hg_2SO_4 " " "	.03942 "		.001619
" " H_2O " " "	46.455 "		52.537
" " H_2SO_4 " " "	.259 "		.0537
D_{25} 1.49037	E. m. f.....	1.42016 V.	
λ_{25} .... .04500	Adjustable cell.....	1.42015 "	

Experiment 13, system without acid added, rotated 55 days, 25°.

Mass of solution in 49.0856 c. cm.....	73.288 g.		Moles in a liter.
" " $ZnSO_4$ " " "	26.637 "		3.361
" " Hg_2SO_4 " " "	.04108 "		.001687
" " H_2O " " "	46.610 "		52.710
D_{25} 1.49308	E. m. f.....	1.42047 V.	
λ_{25} .... .04141	Adjustable cell.....	1.42043 "	

In the present state of our knowledge of solutions, solubility results are best expressed as parts or moles to 100 g. of solvent. but in the present case the solvent water was determined by difference and included all the other errors, while the volume of the solution analyzed was so accurately known it seemed best to calculate the results to moles in a liter, and this data is found in the last column of tables. The character of the curves obtained were the same which ever way the observations were calculated.

It is well known that acid decreases the e. m. f. of standard cells, and this may be seen at once from the tabulated results. On plotting the electromotive forces as a function of the acid con-

centrations, the curve proved to be a straight line, and in the case of the Clark cell the most probable curve for the four determinations showed that a change of one millivolt in the e. m. f. of the cell corresponded to a change of .121 moles in a liter of the sulphuric acid. In the case of the cadmium cell the linear function applies only for concentrations greater than .1 molar acid, since for very small acid concentrations the e. m. f. curve changes are very great and follow quite different curves, but for the region of .1 molar sulphuric acid and beyond a change of one millivolt in the e. m. f. of the cell corresponds to a change of .410 moles in the acid concentration of the electrolyte, so sulphuric acid has a much less effect on the e. m. f. of the cadmium cell than in the Clark cell.

On inspecting the tables it is seen that the rotation does not effect the e. m. f. of the Clark cell; in experiment 13 the rotated cathode system of the Clark cell had the same potential difference as did the unrotated cathode system of the adjustable cell. This is also true in experiments 10, 11 and 12, where acid had been added, also in the case of the cadmium cell, where sufficient acid had been added there was no essential difference between the rotated and unrotated cathode systems, as was found in experiments 1, 2, 3, 4 and 5. But in experiment 6, with only .0153 moles of sulphuric acid to the liter of electrolyte, the rotated system was .00012 volts higher than the unrotated adjustable cell, and in experiments 7, 8 and 9, where no acid was added, the observed increase in e. m. f. was from .00070 to .00189 volts. These high values did not remain constant when the rotation was stopped, but decreased on standing. The first readings were generally taken in about a minute after stopping the rotation and then at intervals. On plotting these observations it was possible to get the value at the moment of stopping the rotation. For example, in experiment 9, one of the last observations made was as follows:

1½	minutes	after	stopping	the	rotation,	e. m. f.	1.0200
2½	"	"	"	"	"	"	1.01992
6⅙	"	"	"	"	"	"	1.01977
24	"	"	"	"	"	"	1.01968

and this indicated 1.02015 as the initial value with an error probably not greater than .00005. That this was the true value was

shown by removing some of the clear superantant liquid and testing it an a clean mercury surface[14]. The reason for the decrease on standing will soon be given.

It was expected that sulphuric acid would decrease the solubility of the sulphates in these systems and, for a given temperature the solubility of the mercurous sulphate and cadmium sulphate (or zinc sulphate) should decrease uniformly with increasing acid concentration, and on plotting the results this was found to be true for all *except the mercurous sulphate in the cathode system of the cadmium cell,* the results are represented by curve 1, Fig

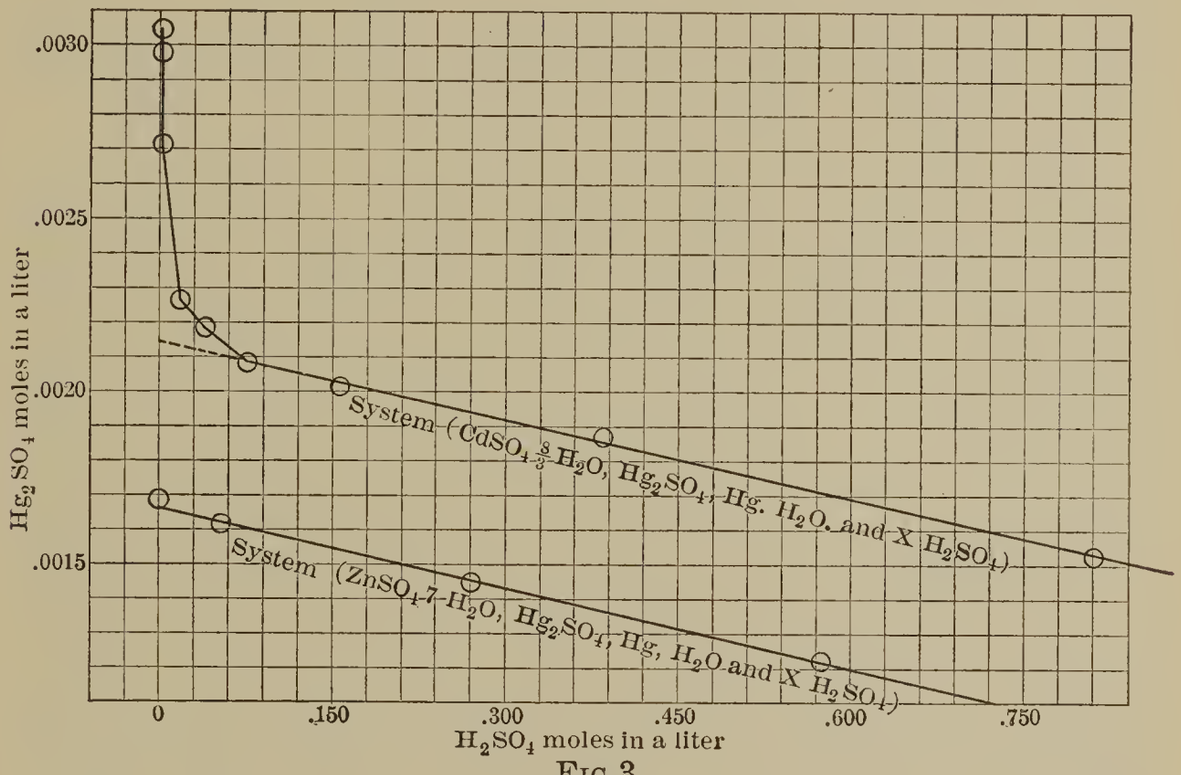

FIG. 3

3. For a concentration of .810 molar sulphuric acid in the system the mercurous sulphate was .001524 moles in a liter; and as the acid concentration decreased the mercury concentration increased uniformly until the acid had been reduced to .08 moles in a liter. The results between these values gave a straight line which was extended to zero concentration of acid. The mercurous sulphate found for the .0384 molar sulphuric acid was distinctly above this line by 4.5 per cent. of its value while for the concentration of .0153 acid in the system the mercury was 7.3 per cent. too high, and for the systems without acid were some 35 per cent. greater than the normal solubility obtained by exterpolating the

[14] Phys. Rev., **25**, 27.

curve. In short, there is a distinct break in the solubility curve of mercurous sulphate in the cathode system of the cadmium cell, and as soon as the sulphuric acid concentration has been reduced to .08 moles in a liter, and this indicates a change in the nature of the solid mercurous sulphate or that a chemical reaction begins and takes place below this concentration. From the well known behavior of mercury salts there can be no hesitation in concluding that a saturated solution of cadmium sulphate hydrolyzes mercurous sulphate. It is also evident that this change in the solubility curve of mercurous sulphate cannot be due to the cadmium sulphate since the solubility curve of this salt in the system increased uniformly with decreasing acid concentration without a suggestion of discontinuity at .08 moles of sulphuric acid.

When mercurous sulphate comes into a cadmium sulphate solution it begins to dissolve and to hydrolyse, and instead of the mercury concentration in the solution becoming constant, when the system is saturated in respect to mercurous sulphate, this concentration keeps on increasing as the slow hydrolysis yields basic salt and acid. In time, the solution becomes saturated in respect to this basic salt, which then appears as a solid phase. But the hydrolysis will not stop at this point until the acid formed by the reaction has reached the equilibrium concentration of .08 moles in a liter. Now the basic salt has a very small solubility, less than the normal salt, which is only .00214 moles in a liter; so the acid concentration was very far from the equilibrium concentration when the basic salt appeared as a solid. This state of affairs at once explains the seemingly abnormal behavior of the cells in which mixtures of basic and normal salts were used as depolarizers[15]. It was expected that any mixture of basic and normal salt which contained an excess of both salts, would give a definite concentration of mercury for each temperature and thus a definite reproducible value to the cells. But they did not agree, and all showed a decreasing e. m. f. indicating that the cathode systems were not in equilibrium. The explanation that seemed most probable at that time was as follows: The basic salt added to the system had been formed by the action of water on mercurous sulphate, it was possibly not the same as that

[15] Phys. Rev., 22, 328.

formed in a cadmium sulphate solution and was therefore ineffective in stopping the hydrolysis. Now it appears that the basic salt was quite incapable of stopping the hydrolosis, and since the necessary acid concentration was lacking the behavior of these cells is readily understood. Cox[16] concluded that there is but one basic mercurous sulphate, and the behavior of these cells may now be brought into harmony with this view. We may then write the reaction as follows: $2Hg_2SO_4 + 2H_2O = Hg_2(OH)_2Hg_2SO_4 + H_2SO_4$, and it is possible to get some idea of the extent of this hydrolysis in the rotation experiments. In none of these experiments was the maximum mercury concentration the point of saturation, of normal and basic salt attained, so we may conclude that the basic salt formed was all in solution, and the increase in the mercury concentration during the rotation is a measure of the amount of hydrolysis. In experiment 9, the analysis showed .003047 moles of mercurous sulphate in a liter, while the normal solubility of mercurous sulphate in a cadmium sulphate solution may be taken to be .00214 miles in a liter, as seen from curve 1, Fig. 3. The difference, .0009 miles, represents the amount of hydrolysis, and according to the reaction a mole of mercurous sulphate gives only ½ mole of sulphuric acid on hydrolysing, so the acid concentration in this system was only .00045 moles of sulphuric acid in a liter, an amount that could readily escape detection by any ordinary means; and we see at once why the attempts to find acid in the rotated systems or old cells have failed. Attention is called to the small amount of this hydrolosis in a cadmium sulphate solution, even under the most favorable conditions in the rotation tubes had only produced .00045 moles of sulphuric acid in three weeks, where .08 were required before equilibrium was established. This .0009 represents the total change in the rotated system; if the reaction was largely at the mercury surface, the rate may have been fairly rapid at that point, but we will first consider the peculiarities of the cadmium cell.

Lord Rayleigh, in 1884, suggested that impurities in the mercurous sulphate used as depolarizer were responsible for observed variations in the e. m. f. of Clark cells; and this was later emphasized by Swinburn[17], while Jaeger and Lindeck[18] came to the

[16] Z. Anorg. Chem., **40**, 178.
[17] Brit. Association Report, 1901.
[18] Z. Instrk., **21**, 33.

same conclusions about the cause of the variations in the e. m. f. of cadmium cells, but there was no suggestion as to the nature of the suspected impurity. Some idea as to the nature of the impurity was first had when precautions were taken to avoid the formation of basic mercurous sulphate in preparing the depolarizer and when the specifications for making the cell were so altered as to avoid hydrolysing the mercurous sulphate in making the paste. Then it was found that the cells had much greater reproducibility and especially that the high preliminary values and subsequent decrease in e. m. f., characteristic of cells constructed in the old way, had disappeared. These results pointed to the basic salt and hydrolysis as the disturbing elements. The first cells constructed according to these principles were the four (D) cells made in December, 1903, and described, together with the method of construction, in a paper by Carhart and Hulett read at the Washington meeting of the American Electrochemical Society, April, 1904[19]. In February, 1904, twelve (F) cells were made, and the agreement among themselves and with the (D) cells indicated a reproducibility of one part in 100,000. But in time deviations in the values of these cells were observed, and in October, 1905, when more cells were made according to the same specifications, and they had a value about one part in 10,000 greater than the older D and F cells. Since then cells have been made at intervals; they have all been kept in an oil bath which very seldom varied .01° from 25°. Some 120 cells have been made according to these specifications during the last five years; some like the D and F cells showed a slow decrease, others have remained constant, and again others have a marked and steadily decreasing value and have become so low that they have been rejected as entirely worthless. Considering only the cells which have not been rejected, a comparison at the present time shows values ranging from 1.01848 to 1.01827 v., the older ones being the lowest. Cells have been made at the Bureau of Standards (Bull. Bureau. of Standards 4.1 and at the National Phys. Lab.[20]) according to the principles just indicated. Some of the cells described by F. E. Smith, l.c. 400, show unmistakably this decreasing e. m. f., and the author was

[19] See also Trans. Amer. Electrochem. Soc., **5**, 59. Trans. International Electrical Congress St. Louis, Vol. **2**, 109-126. Z. Phys. Chem., **49**, 483.

[20] Philos. Trans. A., **207**, 400.

unable to assign a reason for the observed changes, but observed discolorations in the paste of some of these "abnormal" cells, a phenomenon which has appeared in some of our cells which have fallen to a very low value.

In interpreting the observed changes in the e. m. f. of these cadmium cells, account must be taken of the conditions in the cell; the viscous solution and fine grained paste so retard diffusion and convection that concentration differences persist and play a decided rôle. It is the layer of solution immediately next to the mercury electrode and its mercury concentration that determines the potential difference in the cathode leg. That this layer in the cadmium cell is different from the rest of the electrolyte in the cathode leg is shown by the rotation experiments. When the system was rotated the solution became of uniform concentration throughout, and the e. m. f. observed immediately on stopping the rotation gave the potential between mercury and the solution, but as soon as the "paste" settled down on the mercury the potential began to decrease, rapidly at first, and then more slowly. The clear supernatent liquid, however, when brought onto a clean mercury electrode, still continued to show the original high potential against mercury[21], and thus it was seen that only the mercury concentration in the surface layer that had changed.

All of these facts may now be readily interpreted by making a single assumption, namely, that the *rate* of hydrolysis of mercurous sulphate is accelerated by a plain mercury surface. Basic salt and acid would then form more rapidly in the surface layer than in the rest of the electrolyte, and owing to the difficulty of diffusion from this layer it would rapidly become saturated and begin to deposit the basic salt, and with the increasing acid concentration in the surface layer, the mercury concentration passed at once into the descending part of curve 1, Fig. 3.

Thus it is that only decreasing electromotive forces are observed in the cadmium cell, and in the rotated system the high values obtained began to decrease as soon as the solids settled down on the mercury and retarded the diffusion of the acid from the surface layer, and this layer came at once into the descending part of the curve. Stirring the paste at once brought back the high e. m. f. and the true potential between the mercury and the

[21] Phys. Rev. **25**, 27.

system. As soon as hydrolysis was prevented by the addition of sufficient acid, these changes entirely disappeared from the cell and the rotated system, and this fact has defeated all attempts to explain the phenomena from the standpoint of oxidation and reduction or as due to surface tension.

The normal solubility of mercurous sulphate in a cadmium sulphate solution was found to be .00214 moles in a liter, and no doubt the solution in the paste contains a somewhat greater concentration than this. When saturated with both normal and basic salt, the concentration is not far from .004 miles of mercurous mercury in a liter, so it is seen that the surface layer has only a short distance, in quantity, to go to enter the descending part of the curve, and the decrease then depends on the amount of the acid generated in the surface layer and the effectiveness of the paste in preventing its diffusion. It is at once seen that diffusion will not cease until the whole system has reached the equilibrium concentration of .08 miles of sulphuric acid in a liter, and, as this does not take place even under favorable conditions, hydrolysis does not cease, and the basic salt continues to form at the expense of the normal salt, and this change continues until the normal salt has all disappeared from the vicinity of the electrode and there is the possibility of the surface layer to approach the very low mercury concentration due to the basic salt, although there is still plenty of normal salt in the paste.

Furthermore, there is evidence that the basic salt $Hg_2(OH)_2Hg_2SO_4$ does not remain intact[22], but suffers further hydrolysis to mercurous oxide and acid. The mercurous oxide is very unstable, yielding mercuric oxide and oxygen and the conditions for the formation of the very insoluble basic mercu*ric* sulphate, a bright yellow compound. The yellow coloration, noted by Smith[23] and confirmed by the appearance of some of our cells which have gone to a very low value, has been taken to indicate the presence of this basic mercuric sulphate, as the basic mercu*rous* sulphate is colorless or gray[24]. It is quite within the possibilities that these insoluble basic sulphates separate out as incrustations on the mercurous sulphate and cadmium sulphate crystals in the vicinity of the electrode and in this way decidedly

[22] Z. Phys. Chem., **49**, 491.
[23] Trans. Roy. Soc., A., **207**, 407.
[24] Z. Phys. Chem., **49**, 491.

affect equilibrium conditions controlled by these salts. It is true that only a certain per cent. of cadmium cells fall to the very low values, and the number increases with the age of the cells. It is not surprising that the phenomenon does not appear in all cells, no more so than the fact that supersaturated solutions, as near alike as we can make them, do not all crystallize. The cause is possibly the same in both cases, as the basic salt need not appear as solid phase until the solution is supersaturated in respect to them.

Some cells made in 1904 now become of interest. In order to make cells with a lower internal resistance, a coarse grained paste only 3 or 4 mm. deep was used. The mercurous sulphate was formed electrolytically on a large mercury anode in sulphuric acid and, with the low current density used, these crystals were several days in forming. Ten of these (N) cells were made on November 29, 1904, and their record for the first four months follows:

Cell	Nov. 30.	Dec. 7.	Dec. 12.	Jan. 10.	Feb. 1.	Mar. 1.	Apr. 4.
N1.....	1.01842	1.01843	1.01841	1.01839	1.01835	1.01822	1.01776
N2.....	44	43	41	45	41	46	830
N3.....	44	41	40	40	35	26	797
N4.....	44	42	40	42	39	34	797
N5.....	43	41	40	44	41	39	808
N6.....	43	42	40	41	41	40	822
N7.....	44	41	40	40	38	37	812
N8.....	43	45	40	36	32	37	828
N9.....	43	40	40	..	31	19	789
N10.....	43	43	45	45	42	40	824

These cells were found to be in excellent agreement after two weeks, but soon began to show irregularities, and in four months were so irregular and low that observations were discontinued on all but N9 and N10.

	1905			1906			
	June 2.	Nov. 12.	Dec. 14.	May 12.	July 7.	Aug. 14.	Oct. 2.
N9.....	1.01766	1.01715	1.01722	1.01600	11.01462	1.01364	1.01228
N10....	.....	1.01815	1.01816	1.01813	1.01813	1.01812	1.01810

It is interesting to notice that the first comparison of these cells, soon after they were made, showed values if anything slightly higher than the "normal" value of the cadmium cell, so it is to be concluded that coarse crystals and a small amount of them in the paste were nevertheless able to saturate the solution, and in a

comparatively short time, but the conditions were very favorable for diffusion to take place and for the changes just outlined to come into play with the results we would now expect. It was these cells particularly that suggested that attention was to be given to the depth of the paste[26] in the cadmium cell.

H. v. Steinwehr[27] found that by grinding the mercurous sulphate to a fine powder that an increased potential was produced against mercury in a cadmium sulphate solution, and he at once concluded that all variations of standard cells were due to the size of grains of the depolarizer. v. Steinwehr used a large grained mercurous sulphate and selected those of a uniform size, but the cells were irregular and inconstant, and the author attributed their behavior to the little crystals which grew on the larger ones. From the facts now at hand it is to be concluded that surface tension had very little to do with v. Steinwehr's experiments. Not only is the effect of the size of particles not to be feared in constructing the cadmium cell, but it may be concluded that a rather fine grained paste of good depth would give the most constant results in the cadmium cell. The old way of washing the mercurous sulphate with water made for constancy in this system, as the wash water, which was acid, was not entirely removed in making the paste. That such cells do remain constant is shown by the results just published[28] by Prof. Carhart on his (B) cells which were made in 1903, and have been constant for over four years. Reproducibility should be obtained by preparing the mercurous sulphate in a definite way, and with a fine grained preparation made to a paste in the old way the cadmum cells would not show the changes now encountered. It is questionable whether the new method of construction gives a sufficiently greater reproducibility to the cadmium cell to justify the attempt to exclude the basic salt from the depolarizer. If the basic salt is formed in the cell there is no point to many of the precautions now taken.

If we start with mercury, mercurous sulphate and a cadmium sulphate solution, there is little hope of bringing the system to equilibrium as shown by the rotation experiments and subsequent analyses. The equilibrium might be approached from the other

[26] Phys. Rev., 23, 174.
[27] Z. f. Instrumentenk., 25, 205, and Z. Electrochem., 1906, 578.
[28] Phys. Rev., 26, 124.

side if we could start with the components of this system—Hg, Hg_2O, $CdSO_4$ H_2O, SO_3 and H_2O—but the instability of the mercurous oxide excludes this possibility. A knowledge of the equilibrium conditions of this system suggests one further possibility: We may start with a sufficiently acid solution of cadmium sulphate to entirely prevent all hydrolysis of the mercurous sulphate, and then the system rapidly comes to equilibrium; it is a non-variant system when the temperature is fixed and does not show the changes of the cadmium cell. Some 30 of these "acid" cells have been made during the last three years, but the latest results show that the acid concentration is too small, but none of them have shown a tendency to decrease. One cell was made in October, 1905, with .2 molar sulphuric acid, and has shown a constant value of 1.01792 volts and has not varied over .00003 V. at any time during the three years; at present its value is 1.01793. The only defect so far noticed in these cells is a tendency of the amalgam to slowly generate hydrogen, about as much as the zinc amalgam does in the Clark cell. There appears to be no way of keeping the acid concentration automatically fixed, and this may not be necessary, as the cells show no definite evidence of increasing e. m. f., which would result from decreasing acid concentration. In these acid cells a decrease of .004 moles in a liter of the acid concentration would be needed to increase the e. m. f. .00001 volt. These acid cells will be further investigated, for it is evident that the action of the amalgam on the acid solution is a very slow one, and the device of using an acid solution for the unsaturated secondard standards has given most satisfactory results[29].

The results obtained from the experiments (10, 11, 12 and 13) with the cathode system of the Clark cell are plotted in Fig. 3, curve II, and it was seen that there is an entirely different state of affairs in this cell. The mercury concentration in the Clark system to which no acid was added, represented a solubility only slightly greater than that indicated by the exterpolated curve from the results with the Clark systems with acid. The results embodied in curves I and II, Fig. 3, independently confirm the rotation experiments and justify the conclusions that were drawn from those experiments, namely, that a cadmium sulphate solu-

[29] Phys. Rev., 27, 33.

tion hydrolyses mercurous sulphate, while a zinc sulphate solution does not. Now, hydrolysis is of very common occurrence and need not necessarily affect either the reproducibility or constancy of a cell, but in the case of the cadmium cell the hydrolysis establishes a state of unstable equilibrium, and so, sooner or later, depending upon circumstances, changes appear, and it is to be concluded that we are dealing with a non-reversible electrode, a point that has now been pretty thoroughly tested.

Reversibility of the Cathode Systems.—In 1905 an attempt was made to make mercurous sulphate electrolytically in a saturated cadmium sulphate solution instead of in sulphuric acid. It was expected that this method would obviate many of the troublesome operations of making the paste, and produce a product which would necessarily be in equilibrium with a cadmium sulphate solution; but the cells made up with these preparations as depolarizers had a high value and did not remain constant but decreased, as may be seen from the published results[30], and it may be added that they have since decreased. These results were most puzzling at the time; they showed that with rapid stirring and a low current density the mercurous sulphate formed at a mercury anode in a saturated cadmium sulphate solution, was not in equilibrium with the solution and the reaction was not a reversible one, while all the experiments with the Clark cell indicate that its cathode system is reversible. An attempt has been made to directly compare the two cells from this standpoint of reversibility. Both Clark and cadmium cells were made without depolarizers—that is, the mercury cathode was covered with zinc, coarsely crushed zinc sulphate crystals and the saturated solution, while the anode was of the usual construction. The cadmium cells were made in the same way, and were of the same pattern and dimensions. These cells were fastened to a motor driven shaker so regulated that the mercury of the cathode and the materials above it were in constant and uniform motion, then these cells were all charged with the same current, .020 amp. to a cm^2 mercury surface. The mercurous sulphate formed soon saturated the solution and then separated as a solid and was mixed with the crystals of zinc or cadmium sulphate. The motion and low current density offered favorable conditions for forming

[30] Phys. Rev., 23, 175.

the normal sulphate; after about .100 mg. had formed in each cell the charging current was broken and the cells placed in the 25° bath. The paste formed in this way was white in color and about 4 mm. deep. Two cadmium cells were made together in this way, WN1 and WA; the first one was neutral, while WA had been made up with .01 molar sulphuric acid. Later WN2, a neutral cadmium cell, was made in the same way, and the two Clark cells, S1 and SH, were made up without acid and then the depolarizer formed in the cell electrolytically.

Cell. Date.	Jan. 6.	Jan. 9.	Jan. 20.	Feb. 6.	Feb. 18.	Mar. 5.	Mar. 12.
WN1, Jan. 5....	1.01992	1.01974	1.01926	1.01861	1.01827	1.01755	1.01675
WN2, Mar. 12...	.....	.....	.....	.....	.....	.....	1.01890
WA, Jan. 5....	1.01903	1.01883	1.01873	1.01826	1.01828	1.01828	1.0
S1	.....	.....	.....	1.42082	1.42084	1.42086	1.42080
S	.....	.....	.....	80	88	89	88

	Apr. 8.	May 3.	June 6.	June 30.	July 14.	Aug. 18.	
WN1	1.01190	1.9705	1.9644	.9531	.9255	.....	.....
WN2	1.01863	1.01848	1.01770	1.01813	1.01757	1.01611	.....
WA	1.01821	1.01829	1.01831	1.01851	1.01854	1.01859	.....
S1	1.42080	1.42077	1.42071	1.42080	1.42072	1.02068	.....
SH	87	86	79	79	71	70	.....

The two cadmium cells with neutral solutions, WN1 and WN2, were very high at first, and steadily decreased and are still decreasing. There was no suggestion of stopping at the "normal" value of the cadmium cell, which is 1.01840 at 25°, and this emphasizes the conclusions to be drawn from the rotation experiments that this value 1.01840 is the value the cells have when put up in a particular way, and is not an equilibrium value. After the observations of June 6 the pastes in these cells were stirred by shaking the cell, and WN2 particularly at once increased, but only to subsequently decrease. The behavior of WA, the cell made with .1 molar sulphuric acid, is quite different; it had a high preliminary value, indicating that some basic salt was formed. The e. m. f. decreased, but came to a constant value at about 1.01830, and this is about the equilibrium value for cells with this amount of acid. The Clark cells S1 and SH have been quite constant, and the differences between the Clark and cadmium cells is as marked here as is the rotation experiments. These results with the acid cadmium cell and Clark cells suggest that it may be possible to construct these cells and form the depolarizer electrolytically in the cell after they are set up, and

this would entirely avoid the troublesome manipulations and uncertainties incident to preparing the mercurous sulphate and paste.

The Clark Cell.—The Clark cell has been subjected to the most searching tests in the investigations on standard cells, and it is to be noted that the rotated cathode system showed variations of from .00005 to .00010 V., and that the e. m. f. of the rotated system was always slightly greater than that of the adjustable cell which contained the same materials. The analysis of the liquid of the rotated system showed .837 g. mercurous sulphate in a liter of the solution, while the curve 2, Fig. 3, gives as the solubility of normal mercurous sulphate in the cathode system of the Clark cell at 25°, .00166 miles, or .8234 g. in a liter, a measurably lower value. Also, the cells just described, S1 and SH, where the mercurous sulphate was made electrolytically in the cell, are not quite normal, as the e. m. f. is noticeably higher than the normal value for 25°, which is for the Clark cell 1.42040 V. These deviations are all in the same direction, and suggest that a zinc sulphate solution does not entirely prevent a mercurous sulphate from hydrolysing, but this is probably so slight that equilibrium conditions are established before there is a separation of the basic salt, for the severe tests to which this cell has been subjected lead to the conclusion that this cathode system does come to equilibrium and is essentially reversible. The components of this system may then be taken as mercury, mercurous sulphate, zinc sulphate and water, and at a given temperature it is non-varient. But we cannot take the corresponding substances as components for the cadium cell cathode system, for when the hydrolysis is sufficient to produce the basic salt as a solid phase and the acid remains in solution and diffuses to the anode, then these substances are no longer in stoichiometrical relations in the cathode system, and so the products of hydrolysis must be taken as components and the final product due to the interaction with water in the mercurous oxide which is unstable, and the reaction is not reversible. So there seems little hope of obtaining equilibrium with this system, and there probably would not be the possibility of equilibrium in the Clark cell if the basic salt appeared as a solid.

The first Clark cells which were constructed with attention to excluding the basic salt from the depolarizer were the twelve

(H) cells made on March 15, 1904, and at intervals since then cells have been made according to the same principles, and at the present time these cells show no deviations greater than one part in 50,000, and this is the best of evidence that these Clark cells furnish a constant and reproducible basis of electromotive force. Their value has been assumed to be 1.42040 volts at 25°, and they have been the standard of reference for all the work e. m. f. measurements. The temperature coefficient of the Clark cell is about a millivolt per degree, and this was a real objection to the cell before the introduction of thermostats, and was the chief reason for the popularity of the cadmium cells with its much lower temperature coefficient, but for the exact measurements of the present day a thermostat is necessary for any but the Weston secondard cell. The thermostate used for this work is reliable at .01°, and such variations indicate an uncertainty of less than one part in 100,000 of the e. m. f. of the Clark cell. The tendency of the glass to crack where the amalgam contact wire is sealed in has appeared in the older cells and made it necessary to reject many of them. This and other points in the construction of Clark cells may best be considered in a separate article.

Princeton University,
August, 1908.

DISCUSSION.

Prof. W. D. Bancroft: Mr. Hulett's work is so good that one regrets the presence of a few minor verbal infelicities. It is implied that the electromotive force of the Clark cell depends on the temperature only, whereas it is really a function of the pressure and the temperature. Mr. Hulett's statement is strictly accurate only in case his cell has a vapor phase containing water vapor only and neither nitrogen nor oxygen. Since that is practically never the case, it would be quicker and better to mention the pressure, more especially since Nernst once came to grief over this very point.

While it is true that a depolarizer must not be too sparingly soluble, I think that the chief objection to the calomel electrode is that it is not a reversible one. Mr. Hulett implies that it is

merely a result of experience, an empirical fact, that a depolarizer must not be too soluble, and he cites copper sulphate. The real difficulty is quite a different one. The more soluble the depolarizer, the better it is as a depolarizer; but very soluble salt cannot be used at the cathode because it reacts too rapidly with the anode.

These are mere trifles; but there is one point where I think that Mr. Hulett has gone astray in his explanation. Mr. Hulett says that mercurous sulphate hydrolyzes in a cadmium sulphate solution, and that a basic salt appears as a solid phase; but that the hydrolysis does not stop there. Now, this is quite impossible. If there are two solid phases, mercurous sulphate and a basic salt, and if we are dealing with a reversible equilibrium, the concentration of the sulphuric acid cannot exceed the value for a solution in equilibrium with these two solid phases until the whole of mercurous sulphate has disappeared. The presence of the cadmium sulphate does not affect the matter because the solution is saturated with it, and therefore we have one more component and one more phase. I do not question Mr. Hulett's observations in the slightest. It is merely his explanation of the facts which I cannot accept. It may be that he has only one solid phase of variable composition, or it may be that he is not dealing with a system in reversible equilibrium, though this latter seems improbable. One thing is absolutely certain, however, that Mr. Hulett cannot have two solid phases and a reversible equilibrium together with a varying concentration of sulphuric acid.

G. A. Hulett *(communicated)*: Standard cells always have the vapor phase above both the anode and cathode systems, and the pressure of the atmospheric gases in the vapor phase has an effect on the e. m. f. of the cells of the order of 10^{-5} volts per atmosphere, and the slight variations of the atmospheric pressure are entirely negligible; but to be strictly correct one should mention the pressure as Dr. Bancroft suggests. The effect of gravity and surface tension may also be considered when the reproducibility and constancy of standard cells have been sufficiently increased.

In regard to the limits desirable for the solubility of the depolarizers in standard cells, merely a statement of experience

was made without an attempt to consider the reasons for such limitations. Dr. Bancroft thinks the calomel electrode which was cited, is not a reversible electrode, and, if this is the case, it would not be constant. But it is common experience that solutions with small concentrations of mercury show variable potentials with a mercury electrode, and this is possibly due to the action of dissolved oxygen on the mercury. The reaction brings mercury into solutions, and with small concentrations of mercury in the electrolyte a measurable concentration change is produced, but with depolarizers having a solubility of about a gram in a liter this effect is negligible. With very soluble depolarizers, the diffusion to the anode and subsequent reactions produce disturbances as suggested by Dr. Bancroft. Also there results a considerable potential at the junction of the two electrolytes with a very soluble depolarizer, and as this region is subjected to considerable temperature changes in cells there may be a cause of variation at this point.

In considering the hydrolysis of mercurous sulphate by a cadmium sulphate solution (p. 19), the statement was made that the reaction produced a basic salt and sulphuric acid, and when the reaction had produced enough of these substances to saturate the solution with the basic salt that it appeared as a solid phase, but that the hydrolysis did not stop at that point, but proceeded until the equilibrium concentration of the acid was generated. The conclusion was drawn that the basic salt alone was incapable of preventing the hydrolysis, and this was an important part of the explanation of the observed behavior of cadmium standard cells. These statements seem to Dr. Bancroft to be in conflict with well established principles. He contends that it is quite impossible for the hydrolysis to continue after the basic salt appears as a solid phase, if we are dealing with a reversible equilibrium. This objection does not hold good, because the system is not in equilibrium when the basic salt first appears. The facts are that at .08 moles of sulphuric acid in a liter—the concentration at which the break in the solubility curve of mercurous sulphate appeared—there are only .000207 moles of mercurous mercury in the solution, and, if we started with mercurous sulphate and a cadmium sulphate solution, it follows that the basic salt must have separated as a solid long before this acid concentration was

generated, because acid and basic salt are formed in molecular proportions by the hydrolysis. When the hydrolysis had generated enough basic salt to saturate the solution, the salt appeared as a solid phase, and merely limited a further increase in the mercury concentration of the solution, but the system was not in equilibrium, and the hydrolysis continued until the equilibrium acid concentration was formed, and not until this point is reached can the principles, indicated by Dr. Bancroft, be applied.

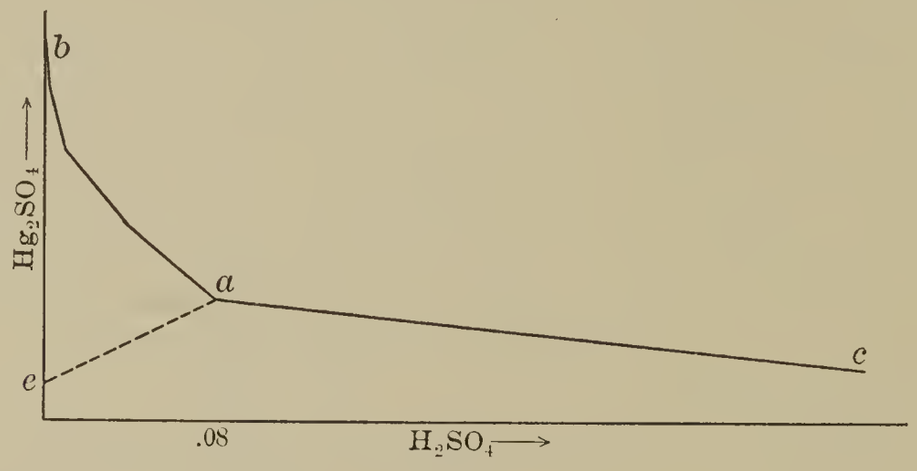

FIG. 4.

Referring to the accompanying diagram, the point (a) at 25° represents the conditions at which normal mercurous sulphate, cadmium sulphate, mercury, solution and vapor can exist in equilibrium. For greater concentrations of acid, the basic salt must disappear, and the curve (ac) is an equilibrium curve showing the solubility of mercurous sulphate in the system with variable amounts of acid. With less amounts of acid than .08 moles of sulphuric to a liter the normal salt must disappear, and the equilibrium curve would be the solubility of the basic salt in the system with varying amounts of acid. This curve would probably take a downward turn from (a), since the basic salt has only a slight solubility. It is clear, therefore, that if .08 moles of acid to a liter is the equilibrium acid concentration for this system, that any system which contains less than this concentration of acid and the normal salt must be in unstable equilibrium. Now, normal salt was always present in excess in all the systems we investigated, and this supports the contentions previously made (Phys. Rev.), as well as the results now obtained, namely, that this cathode system of the cadmium cell is in unstable equilibrium unless at least .08 moles of sulphuric acid are present in

a liter of the solution, and it may here be further emphasized that the part of the curve represented by (ab) in the appended diagram is not an equilibrium curve, but represents the results obtained with systems which contain an excess of the normal salt and rotated until no further change is observed. The reason that equilibrium is not immediately established, or established in any "experimental" time, is considered as due to the slowness of the reaction as pointed out in the article.

A paper read by Dr. H. E. Patten at the Fourteenth General Meeting of the American Electrochemical Society, at New York City October 30, 1908. President E. G. Acheson in the Chair.

THE BEHAVIOR OF THE NICKEL ANODE AND ITS BEARING ON PASSIVITY.

By E. P. Schoch.

The subject of passivity has been, and is still, attracting a great deal of attention, particularly among electrochemists. The first work on this subject was done by Schoenbein and Faraday, while the most recent contributions, such as that of Sackur (1)*, and of Fredenhagen (2), have appeared but a few weeks ago, and the indications are that many investigations on the subject are in progress. Considered particularly with reference to nickel and iron, the phenomenon may be characterized in its outward appearance by the following: Under the influence of certain reagents, or while the metal is acting as the anode in certain solutions it is changed so that its potential is far below that which should ordinarily characterize it. The lowering of the potential may take place to a widely varying extent. At the same time the metal is attacked less than we should expect it to be attacked, in fact the attack may become so slight as to be negligible. The surface may or may not be covered with a layer of oxide, in some cases the surface remains particularly bright. In contradistinction to this passive state of the metal, we recognize an *active* state, in which the metal has a higher potential or is more electro positive and is readily corroded or acted upon by chemical reagents.

The cause of the passive state has been the object of many inquiries. Inasmuch as the subject has been reviewed extensively quite recently in the article of Fredenhagen referred to, a detailed review may here be considered to be unnecessary. A statement of the different theories of passivity together with the lines of attack is probably sufficient.

* The numbers in parentheses after the proper names refer to the list of references given at the end of the paper.

Fredenhagen has classified the theories of passivity as follows: No. 1, the oxide theory of Faraday, according to which the metal is protected by a layer of its own oxide. No. 2, the valence theory of Krueger-Finkelstein and W. J. Mueller; these authors consider the cause of the passivity to be within the metal itself. No. 3, the reaction-velocity theories: (a) the general formulation according to Le Blanc; (b) the special formulation of Fredenhagen, and Muthmann and Frauenberger: these authors consider that the main cause lies in the slight velocity with which oxygen reacts with the metal passivized. Hence the oxygen accumulates and causes a lower potential in a manner similar to anodic polarization, as with platinum.

To this classification should be added under heading 3 above, the theory of Sackur, who considers that the particular process, the velocity of which is so slight as to impede the whole, is the union of oxygen and hydrogen within the body of the metal.

The lines of attack upon this problem have been many, and for an understanding of the problem as it presents itself to-day should be presented in the main, and the results obtained briefly described. Leaving out of consideration the first work on passivity, which is mainly of a quantity nature, the lines of attack have been the following: No. 1, the relation of the current density to the anodic polarization with different electrolytes. No. 2, the measurement of the capacity of the metal under various conditions. No. 3, Observations of the optical properties when no oxide layer is visible to the naked eye. No. 4, the effect of alternating current electrolysis. No. 5, the catalytic influence of the metal upon the union of hydrogen and oxygen gases at ordinary temperatures. No. 6, the conditions under which a metal oxide is formed at the anode. No. 7, the effect of mixed electrolytes as used for the production of Luckow's phenomenon and the quantitative determination of the attack upon the anode by various electrolytes.

The first line of attack has been pursued by many investigators, notably, Finkelstein (3), Fredenhagen (4), Mueller (5), Sackur and Alvares (6), and many others who through the results of their observations added zinc, copper and magnesium and many other metals to the list of passivizable metals. These results show in the main that metals are most readily passivized in

alkali solutions, but many others, notably nickel and iron, are also passivizable in acid solutions. In connection with the latter, it is observed that passivizing appears to take place when a certain critical current density is reached. This critical current density is greater as the concentration of the acid is greater, and as the temperature is higher. With sufficiently concentrated acids, or sufficiently high temperatures, passivizing may not take place at all. The presence of halideions in solutions tends to prevent passivity so that with sufficient concentration of these passivity is never attained. In my experimental work, to be presented below, I have followed this same line of attack, but for reasons given I have considered it necessary to use essentially different electrolytes from those heretofore usd, hence a further discussion on this point may be deferred.

The second line of attack was first tried by Finkelstein on iron, and subsequently again by Gordon and Clark (7). With a different idea in view, namely, to study the anodic behavior of aluminium, Schulze (8) investigated the behavior of aluminum and other metals by practically the same methods. The procedure in the main is that of balancing the capacity of the anode with that of a condenser of known capacity, using at the same time a shunt of variable, known resistance parallel to the condenser. By this means the condenser effect of the insulating film around the anode, as well as the "leak" current through the film, may both be properly balanced and measured. It appears to me that the conclusions reached by Finkelstein and Gordon and Clark are based on experimental facts which may involve unknown factors. Hence they should not be relied upon until they have been more extensively investigated. The measurements of Schulze, however, are conclusive. He found that the "valve" effect of the aluminum anode is due in the main to a layer of oxygen gas, and the layer of oxide which is also present serves merely as a supporting skeleton. Without this supporting skeleton the layer of oxygen could attain only a lesser thickness, and hence be much less effective. When we consider that the condenser effect of the aluminum anode in its outer manifestations is the same in kind, though different in degree, as the polarization effect which characterizes passivity, and when furthermore we consider that "valve" effects and passivity are produced under conditions which

do not differ essentially, then we must realize that Schulze's results indicate that passivity may be caused by a thin layer of oxygen, such as would probably exist if the supporting oxide were entirely absent.

The third line of attack, the attempt to decide by optical measurements whether an oxide film is present or absent when none is noticeable by the naked eye, has not been able to decide the question. Perhaps it is admissible to say that the presence of the oxide film in many cases yet remains to be proven.

The fourth line of attack, the effect of alternating current electrolysis, is one of the most recent lines of procedure that have been directed to this problem. Ruer (9) has studied the solubility of platinum under the influence of the alternating current and has reached the conclusion that the ability of platinum to resist attack is due to passivity. He finds that when he subjects a platinum pole to an alternating current and makes it simultaneously the anode of a direct current with small current density, the platinum dissolves extensively. The same results were obtained when instead of the direct current an oxidizing agent is placed in the electrolyte. He is of the opinion that the platinum dissolves through the following procedure: Under the combined effect of the direct current and the oxidizing agent or that stroke of the alternating current which makes the platinum the anode, a layer of a peroxide is formed. This peroxide is partly reduced by the reverse stroke of the alternating current and is then in the nature of a basic oxide which readily dissolves with the acid of the adjacent layer of the electrolyte. He has actually succeeded in isolating an oxide of platinum which has all the requisite properties.

However, he has not produced a proof that the layer is actually formed under the conditions under which passivity is observed. I shall attempt to show below that the whole phenomenon may be seen to have a better connection when viewed from a different point.

The fifth distinct line of attack was made through a unique observation of Sackur's: he found that a platinum anode polarized with oxygen would oxidize *but slowly* any hydrogen that floats up to it by diffusion. On further investigation of the subject he found that the metal charged with hydrogen would accelerate

the reaction between hydrogen and oxygen far more than when the metal is charged with oxygen. Furthermore, he showed that the catalytic influence upon the union of hydrogen and oxygen at ordinary temperatures varies greatly with different methods. He found there catalytic influence to be in the following order: Silver, platinum, copper, tin, iron, nickel, chromium. This order is in a measure the same as the order of their tendencies to passivity. The assumption that the first step in the anodic action of a metal with an electrolyte is the displacement of hydrogen ions by the metal according to the equation

$$M + 2H^{+} = M^{++} + H_2$$

leads him to the conclusion that the retardation of the oxidation of the liberated hydrogen would retard the whole and hence produce the phenomenon of passivity. Though this idea has not gained much ground—in fact, has not been followed out by the author recently—yet his results demonstrate that oxygen gas may cover a metal and prevent direct contact with the electrolyte. This seems to me to be a very important result.

The sixth line of attack, the conditions under which an oxide is formed at the anode, appears to me to have produced many more results of merit than appear to be credited to it. Mueller and Spitzer (10) investigated this subject very thoroughly, with a view to determine the conditions under which an oxide may be formed at the anode. This work taken in connection with the results of many others and our general knowledge of the subject, makes it very plausible that oxides which appear on the anode are most probably formed *in* the electrolyte, and they are precipitated back upon the electrode rather than formed direct by the union of oxygen with the metal.

The seventh line of attack, the action of mixed electrolytes, is mainly the work of Le Blanc and Levi (11). These authors reached the conclusion that there can be no oxide layer present when passivity is caused in certain electrolytes, notably acid electrolytes, and unless a distinction is to be drawn between passivity in alkali solutions and passivity in acid solutions, the conclusion is reached that an oxide layer is not the cause of the passivity. Haber and Goldschmidt (12) used an experimental procedure similar to that of Le Blanc and Levi and worked essentially with alkaline solutions. They reached the conclusion that the

passivity of iron, which is the only metal they worked with, is due to an oxide film which instead of forming a continuous cover has holes in it here and there.

I was led to the study of passivity of nickel through a study of the reversible potential of nickel, which work I completed this summer and submitted for publication. In this work I ascertained that the reversible potential of nickel is 0.48 volts with reference to the normal calomel electrode as a zero, the nickel being negative to the calomel electrode.* I found that nickel, which occludes hydrogen very readily may show as a result any potential from 0.48 to 0.88 and more in N/1 $NiSO_4$. Furthermore, I found nickel has an exceedingly small ionization velocity. Nickel may also occlude oxygen and then may show any potential below 0.48 volts. Thus exposure to the air lowers the potential to *about* 0.25 volts, and various oxidizing agents will give up oxygen to it, lowering its potential down to an extent depending upon the strength of the oxidizing agent. The same facts hold in regard to iron, the potential of which towards N/1 $FeSO_4$, having been found by T. W. Richards (13) to be 0.71 volts. In the same paper Richards showed that all the higher potentials of iron are due to hydrogen. That air imparts a lower potential to iron is well known, and the direct proof that oxygen is taken up by iron from potassium dichromate has been produced by Cushman (14). These facts define more definitely the active and the passive states: The active states of nickel and iron are primarily due to their solution tension by virtue of which they produce in the solutions of their neutral sulphates the potentials mentioned above, 0.48 and 0.71 respectively. Whereas the active state formerly recognized, which was produced either by dipping these metals in hydrochloric or sulphuric acids and allowing them to evolve hydrogen, or by making these metals cathodes in solutions in which they evolve hydrogen consist of the metals with a lot of occluded hydrogen. This state, due to the occluded hydrogen, plays no part in the phenomenon of passivity and may be neglected in this consideration.

Both nickel and iron exhibit an exceedingly small velocity of ionization which is shown first of all by the "current-density"-

* All potentials given in this paper are to be considered as measured in the same way.

"anodic-potential" curves of these metals as compared with the curves obtainable with such metals as copper, silver, zinc, etc. The curves for the latter metals are practically parallel to the "anodes-potential" axis for moderate current densities when these metals dip in normal solutions of their simple salts, sulphates and chlorides.* Whereas the nickel and iron curves bend away from the potential axis enormously from the start, yet it is safe to assume that if the anodic action in the case of silver, copper and zinc consists in the direct formation of ions from the metal, then the same is true for nickel and iron as long as we consider small current density only. Thus with the current density of 20 milli-amperes per 100 sq. cm., nickel dissolves quantitatively in N/1 $NiSO_4$, according to Farraday's law—a fact which I have ascertained repeatedly by trial—and the pole exhibits during this operation a potential of 0.1 to 0.15 volts. At higher temperatures, the velocity of ionization of nickel is much greater, as may be seen from the accompanying curves for 25 and 95 degrees C. respectively. At 95 degrees, with a current density of 50 milli-amperes the nickel dissolves in accordance with Faraday's law and never show a potential less than 0.42 volts, while its equilibrium potential in the same solution at the same temperature is at most only 0.02 volts higher than this. Thus we see that at higher temperatures the curve for nickel is much flatter, and is quite similar to the curves for copper, silver, etc. If it is considered to be sound to believe that the action with these metals consist of the direct formation of ions from the metals, then the same must be true for nickel. I may mention here that I have done the corresponding experiments with iron, but have recently found a possible source of error in my measurements, so that I do not care to mention the quantitative results here, but I feel safe in saying that the phenomenon with iron is the same as that with nickel. Finally the great difference in the ionization velocity of nickel and iron as contrasted with zinc, silver, etc., is strikingly shown by the behavior of their amalgams: those of zinc and silver will bear a considerable current density without undue lowering of the anode potential, while iron will bear very little current density, and nickel practically none. Yet iron amalgam will readily give the potential 0.71 volts, while the

* Strictly speaking, they are logarithmic curves.

most concentrated nickel amalgam will not form ions rapidly enough even to measure with the capillary electrometer by the well known compensation method.

With these facts before us, let us now consider all the possible actions that may take place at an anode. There may be summed up as follows: No. 1, the direct change from metal to ions. No. 2, the displacement of hydrogen ions by the metal. No. 3, the discharge of oxygen or halides, etc., upon the electrode. No. 4, the union of hydrogen and oxygen, or hydrogen and halogen within the electrode. No. 5, the union of oxygen or halogen within the metal. No. 6, the neutralization and solution of an oxide formed according to No. 5, by means of a free acid adjacent to the electrode. In the following we will leave halides out of the consideration for the present and confine our attention to what would happen in say sulphate solution.

One or several of these changes may predominate under one condition, and under another may sink into the background: hence, it is advisable to ascertain, if possible, which of these changes takes place under different conditions. The mode of investigation employed by Fredenhagen and Sackur, namely, electrolyzing with rather strong acids and observing the potential with different current densities appears to me to be not well chosen, because the conditions are not securely responsible. The system is not in equilibrium, but very far from it, and the gradual change from the point of equilibrium has not been carefully shown. I thought it best to begin the study at the point of the equilibrium potential of nickel. Since nickel reacts with acids evolving hydrogen, it can be in equilibrium with absolutely neutral solutions only. Thus neutral nickel sulphate was the first electrolyte tried.

My work on the nickel potential had shown me the necessity of removing all gases such as hydrogen and oxygen out of the electrolyte, and out of the metal before attempting to obtain the potential. I found hydrogen very difficult to remove, in fact, I could remove it only by allowing the pole to stand immersed in the neutral electrolyte for one or more weeks. Oxygen, however, could be removed fairly readily by heating the electrolyte with the pole in it to boiling in a vessel so fitted with a stopper and a glass tube, that after boiling out all the oxygen or air,

then on cooling the solution could finally be drawn into the flask to fill it when the steam condensed. The nickel poles had to be carefully sandpapered because in the event that any nickel oxide had formed on its surface it became practically impossible to remove the last traces of oxygen even by protected boiling--

The current densities that nickel can support in $\frac{N}{1}NiSO_4$, without dropping its potential into the region of passivity are so small that convection currents carrying hydrogen from the cathode to the anode may completely vitiate the results: that is, the currents obtained are of the dimensions of the well known "rest current," and hence the possibility of producing hydrogen in the electrolyte had to be guarded against. For these reasons the experimental arrangement adopted was the following: A large test-tube, 2 inches diameter and 8 inches in length, was fitted with a rubber stopper, through the center of which was passed a narrow porous tube extending into the test tube. This porous tube was closed at its lower end with a rubber stopper; and tightly fitted through the center of this stopper was passed a glass tube extending through the entire length of the porous tube. A glass rod of the size which fitted closely inside the glass tube just mentioned extended down through this glass tube, and it was bent up around the lower end of the porous tube so as to extend up inside of the test-tube almost to the rubber stopper A piece of sheet nickel was cut to fit closely inside the test tube with a lip extending through the rubber stopper. A piece of lead pipe, the surface of which had been thoroughly converted to lead peroxide was inserted around the central glass tube and inside the porous tube. This served for the cathode, and by this means the evolution of hydrogen was absolutely avoided, the porous tube in turn serving to prevent the contamination of the liquid outside the porous tube with lead. The sheet nickel anode had a surface of 270 sq. cm. Into the stopper was also fitted a syphon tube connecting with a flask clamped beside the test-tube. A tube drawn to a fine capillary, the end of which was placed against the nickel electrode served to make connection with a normal calomel electrode, the connecting tube being suitably bent so as to prevent the mixing of liquids by convection. A thermometer also was inserted. It will be noticed that the

space in which the nickel electrode is placed is absolutely closed to the outside except through the space between the stirring rod and the glass tube in which it turns. To lessen this opening, a rubber tube was slipped over the end of the glass tube, a portion of which closed against the stirrer. The flask into which the syphon extended was placed at such a height that the level of the liquid in it was well above the stopper of the test tube, and in the event of a leak anywhere, air would not have been admitted, but liquid would have been forced into the test tube. The stirrer was turned at a rate of about 200 revolutions per minute.

For any one determination, the test-tube was completely filled with the electrolyte to be tried and a sufficient excess of the electrolyte was put into the flask into which the syphon was extended. The liquid in the test-tube as well as the extra liquid in the flask, were heated to boiling, and after boiling vigorously for several minutes the temperature was slightly lowered so that the steam was condensed, and the test-tube and syphon would fill completely with the electrolyte. If care has been taken not to contaminate the sheet nickel with hydrogen of oxide, then the potential of this nickel electrode in neutral normal nickel sulphate will be about 0.42 volts at 95° C. and 0.48 volts at 25° C.

Different current densities were applied to this electrode, and after a sufficient length of time had elapsed so that a permanent condition was reached, then the potential of the anode against the normal calomel electrode was recorded. The current densities ranged from 0.04 milli-amperes to 0.5 amperes per 100 sq. cm. The permanent condition was reached in a few minutes when the potentials were only slightly below the equilibrium potential, e. g., differed not exceeding 0.05 to 0.08 volts. With larger current densities which produce a greater drop in potential it became very difficult to reach a permanent state that could be duplicated. Hence, some of the results are correct to within 0.05 volts at best, though most of them may be considered as being much nearer the probable correct value.

The following results are obtained with neutral normal nickel sulphate. This solution was made absolutely neutral by adding enough dilute sodium hydroxide solution to produce a permanent precipitate. It was filtered before it was used.

1. $\frac{N}{1}$ $NiSO_4$—neutral, at 26° C.

Current Density per 100 sq. cm.		Anode Potential	
.0		.48	volts.
0.04	milli-amperes	.47	"
0.08	" "	.463	"
0.12	" "	.457	"
0.2	" "	.445	"
0.4	" "	.432	"
0.8	" "	.42	"
2.0	" "	.389	"
4.0	" "	.358	"
8.0	" "	.315	"
12.	" "	.27	"
16.	" "	.222	"
24.	" "	.125	"
32.	" "	.05	"
36.	Here passivizing began.		

In order to facilitate the oversight of the results they will all be given in the form of plots (which see) and I will confine myself here to pointing out the main features to be noted.

The curves presented herewith give the polarization values of the nickel anode in the following electrolytes:

I.—N/1 $NiSO_4$ at 26° C.
II.—N/1 $NiCl_2$ at 26° C.
III.—N/1 $NiSO_4$ at 95° C.
IV.—N/1 $NiCl_2$ at 95° C.
V.—N/1 $NiSO_4$ with N/100 H_2SO_4.
VI.—N/1 $NiSO_4$ with N/10 H_2SO_4.
VII.—N/1 $NiCl_2$ with N/10 HCl.
VIII.—N/1 K_2SO_4.
IX.—N/1 $NiSO_4$, using the anode just after it had been used with N/1 K_2SO_4.
X.—N/1 K_2SO_4 with N/10 KOH.
XI.—N/10 KOH.

It is seen that Curve V slopes away decidedly from Curve I, and Curve VI still more so, whereas Curve VII practically coincides with Curve II. Since the electrolyte for Curve VII (*i. e.*, N/1 $NiCl_2$ with N/10 HCl) has no lesser concentration of hydrogen ions than the electrolyte for Curve VI (N/1 $NiSO_4$ with N/10 H_2SO_4), it appears that the hydrogen ions cannot act

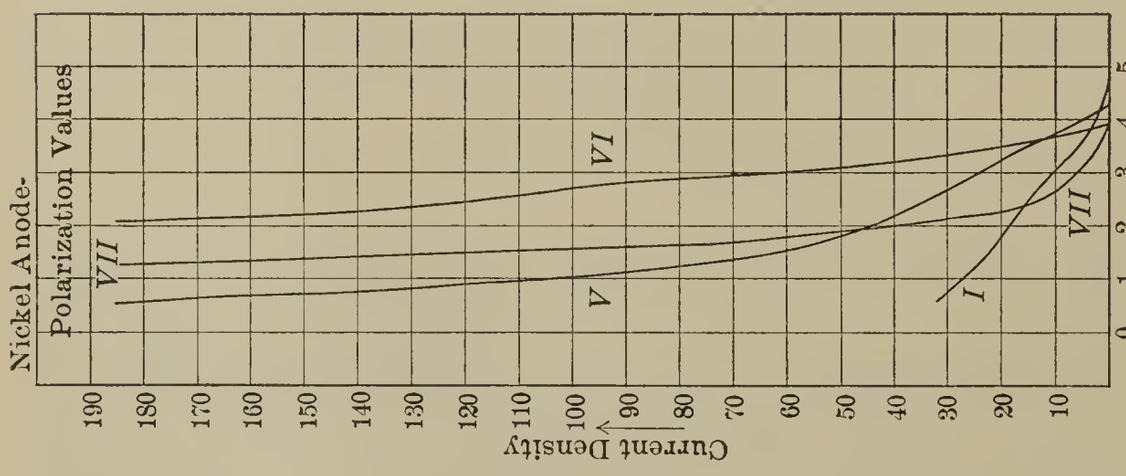

FIG. 1.

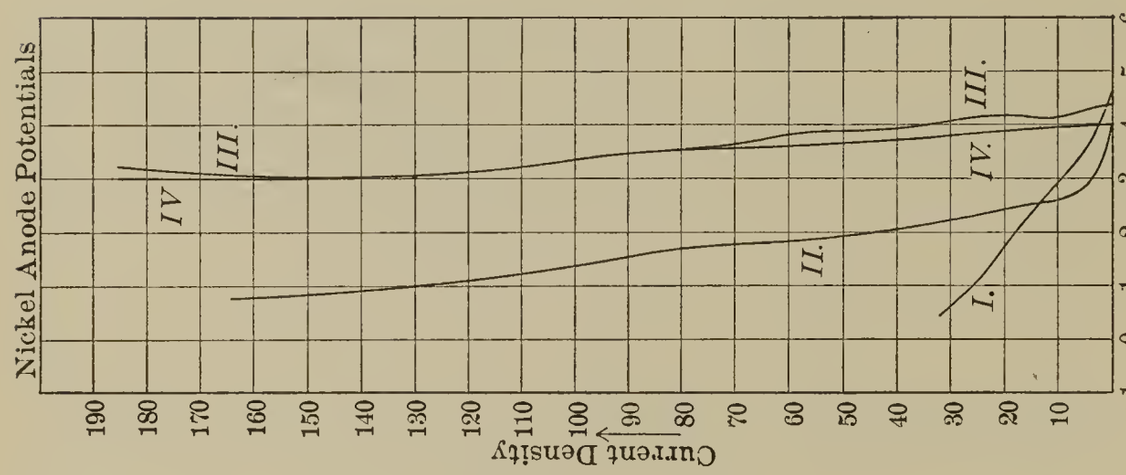

FIG. 2.

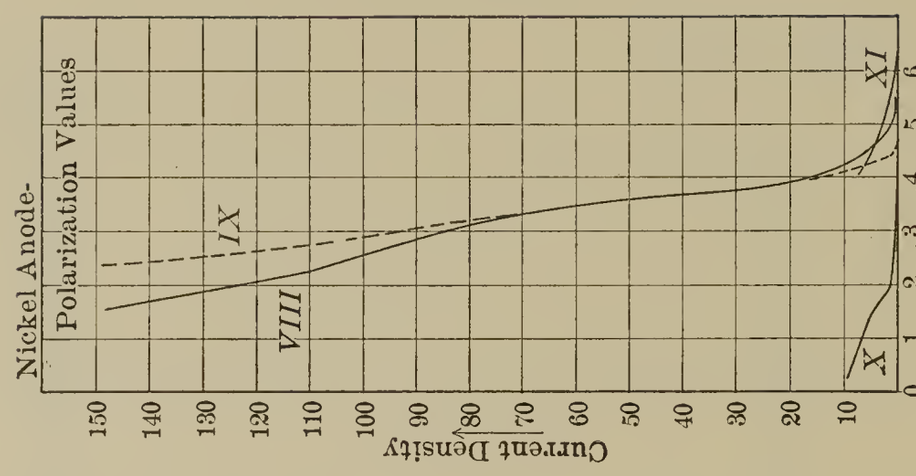

FIG. 3.

directly to influence the behavior of the nickel anode. Since furthermore Curves X and XI slope away strikingly on the opposite side from Curve I (or rather, from its correspondent, Curve VIII—concerning which see foot-note), it appears that the concentration, more specifically, the discharge of the hydroxyl ions is the prime factor which produces the normal polarization effects that lead to passivity. The influence of hydrogen ions is thus seen to be indirect, and due to the fact that the concentration of the hydroxyl ions is less in inverse proportion to the concentration of the hydrions.

From results gathered in connection with these electrolyses as well as from my results on the reversible behavior of the nickel electrode which will appear elsewhere, I am lead to believe that the behavior of the nickel anode in sulphate electrolytes is the resultant of two effects, one, the direct ionisation of the metal; the other, the simultaneous discharge of oxygen. Nickel appears to have a very small ionisation velocity and hence cannot furnish ions fast enough for any largere current densities. Hence the passage of larger currents takes place partly by the discharge of oxygen. It appears that the areas thus covered with oxygen are not able to send out ions, and thus the *active* area is decreased as the discharge of oxygen is increased either through greater current densities or through greater concentration of hydroxyl ions. When the electrolyzing current is discontinued, the discharge oxygen tends to reionise, and forms with the active spots a short-circuited cell, through the action of which the oxygen is finally removed and the surface recovers its original condition. If the area of the active spots is small compared with the area of the oxygen spots, then during action of the "short-circuit" the potential shown by the active spots (which is the observed potential of the pole) may have any value *below* the potential of nickel, just as any cell when an excessive amount of current is drawn from it shows a voltage far below its normal. In this way the low potentials of passive metals are probably produced.

I found that a nickel surface undergoes a peculiar change in alkali sulphate solutions, as a result of which the polarization curve extends more nearly parallel to the current density axis. However, such a surface gives the same values in N/1 K_2SO_4 as in N/1. $NiSO_4$, as shown by Curves VIII and IX. Thus, for the experiments with alkaline solutions, Curve VIII should be referred to as corresponding to Curve I.

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DISCUSSION.

Dr. H. E. Patten: Dr. Schoch was led into his study of the passivity of nickel and iron through another line of work; he was working on the potential of the reversible electrode.

Prof. W. D. Bancroft: Mr. Schoch apparently defines as absolutely neutral solution, one he gets by adding caustic potash to the nickel cell, and then filtering from the solution. Of course, that would give you a neutral solution only in case there was no hydrolysis nickelous salt, or there was no dissolving of the nickelous hydroxide by the nickel salt.

I ask Dr. Patten whether the possibility of hydrolysis of nickelous sulphate has been considered?

Mr. Patten: The impression I gather is that it had been neglected; that is, the method of getting the solution into a neutral condition was to put in a certain amount of caustic potash, but the objection to this is that when you filter off a solid phase, resulting from the addition of caustic potash to the neutral nickelous sulphate, you change the conditions of equilibrium, and give a chance for hydrolysis to take place.

Dr. Schoch *(communicated)*: The object of using a nickel sulphate solution which had been treated with a small quantity of sodium hydroxide, etc., was to obtain solutions which were absolutely identical in concentration of hydrions or hydroxylions. It is merely one step in making the conditions accurately reproduceable.

A paper read at the Fourteenth General Meeting of the American Electrochemical Society in New York City, on October 30, 1908; President E. G. Acheson in the chair.

THE FORMATION OF NITRIC OXIDE FROM THE AIR BY MEANS OF A CURRENT OF ELECTRICITY OF LOW VOLTAGE

By G. W. Morden.

The following paper includes the results of an investigation carried out in the department of physical chemistry in the Technische Hochschule Karlsruhe, Prof. F. Haber, Director.

The increasing need of nitrates in agriculture and in the industries has produced so great an increase in the demand for Chili saltpetre that the export of this material now amounts to about 1¾ million tons yearly. It has been estimated that the increasing use of this material will accomplish the exhaustion of the deposits now being worked in from 25 to 40 years. Although the Chilian government asserts that the lately discovered deposits exceed in magnitude those which up till now have been the source of supply, yet the figures given show the importance of finding a substitute for these saltpetre deposits. In addition to the Chili saltpetre, the ammonium sulphate that is won as a by-product in the dry distillation of coal is used to a great extent in agriculture as an artificial fertilizer. Four-fifths of the saltpetre exported is used as fertilizer, the other fifth being applied to the manufacture of explosives, sulphuric acid, and to the preparation of dye-stuffs. In order to keep pace with the increasing demand for fertilizers, great exertions have been made to increase the yield of ammonium sulphate from coal. Caro[1] asserts that only one-sixth of the nitrogen content of the coal is obtained by the ordinary dry distillation of coal, but that in the Mond process there is an almost quantitative transformation of the nitrogen to ammonia. This process consists in removing the gas from the coal by means of air and an excess of water

[1] Zeitschrift für angew. Chemie, **19**, 1077 (1906).

vapor. One-third of the total annual nitrogen supply is obtained from ammonium sulphate, the balance from Chili saltpetre. If, however, the total nitrogen of the coal is obtained more than twice the requirement at present supplied by the Chili saltpetre can be covered.

Of the other processes which serve for the winning of nitrogen in a form that can be used by plants, that which utilizes the air as the source is the most important, and by far the most important of these methods, is that which produces cyanamid from calcium carbide and nitrogen, according to the process of Frank and Caro[2], and its modification by Polzenius[3].

Besides this process, the winning of the atmospheric nitrogen by means of the electric current has met with success. In nature this happens at every discharge of electricity through the air in the form of lightning. Similar discharges, as sparks, by means of high tension machines, have not produced such yields as to lead to the development of a commercially successful process. On the contrary, the electric arc which permits the concentration of great quantities of energy in a small space has been used to obtain such concentration of nitric oxide that the product can be profitably worked up.

From the standpoint of economy, the question of the yield per kilowatt hour is the most important, and accordingly the chief aim is to so arrange the arc that this relation shall be the most favorable. A few processes that have a special technical interest may be here mentioned.

In the year 1895, Neville and Guye[4] obtained a patent which is based on the removal of the nitric oxide as quickly as possible from the arc, while at the same time the entering air is brought directly into the arc.

In the year 1899, McDougall and Howles[5] patented a process in which the arc burned in earthen vessels with the electrodes so arranged that the distance between them could be regulated and the gas after being subjected to the action of the arc was led through towers where the nitric oxide was absorbed by water. Eight arcs were arranged in parallel and the energy was

[2] German Patents 108,971, 116,087, 116,088.
[3] German Patent 163,320.
[4] German Patent 88,320 (1895).
[5] English Patent 4,643.

supplied by a 7,500 volt alternating current machine. The yield in the most favorable cases was about 34 gms. of nitric acid per kilowatt hour.

Kowalski and Moscicki[6] with a similar arrangement, but with a tension of 50,000 volts, a current of 0.05 amp., and 6-10,000 alternations per second, obtained a similar yield.

In the United States, Lovejoy and Bradley[7] worked out a process which was patented by the Atmospheric Products Co. at Niagara. They used thin arcs which were continually broken and made by the rotation of the apparatus, so that an extraordinarily rapid removal of the gas from the influence of the arc was accomplished. The direct current was supplied by a 10,000 volt machine to a series of parallel arcs provided with appropriate resistances in the form of inductance coils. The positive electrodes were arranged on the rotating axis and passed the negative electrodes at a distance so short that the arc "struck over." The total current supplied by the dynamo amounted to one ampere, while the current for each arc was about 0.004 to 0.005 amp. The air was admitted to the chamber at both ends and drawn out through openings close to the negative electrodes.

The process of Birkeland and Eyde[8] which is in operation at Notodden, Norway, is, from the commercial standpoint, the most important and the most successful. In this process the electrodes are in series with an induction coil, and the arcs burn between the poles of a strong electromagnet. By this means the arc is flattened and driven outwards from the electrodes till it is broken, being immediately re-made between the electrodes. This electromagnetic "bellows" acts several hundred times per second. A 2,000 kilowatt generator with 5,000 volts tension supplies a three-phase alternating current, and the three ovens which are arranged between the three phases work over 25,000 liters of air each per minute. According to the investigations of an international commission of experts[9], a yield of 500 kilograms of nitric acid per kilowatt year can be guaranteed. With a power price of $5.00 per kilowatt year as given by Klaudy[10], this amounts to 4½ cents per kilogram of combined

[6] English Patent 20,497 (1903).
[7] English Patent 8,230 (1901).
[8] Norweg. Patents 13,240 and 13,280 (1903).
[9] Donath and Freuzel, Die Technische Ausnutzung des atmos. Stickstoffes, p 549.
[10] Zeit. für Electrochemie, **12**, 545 (1906).

nitrogen as nitric oxide, while with a market price of $5.25 per 100 kilograms for Chili saltpetre, the price of the combined nitrogen amounts to about 33 cents per kilogram.

In opposition to the thin arcs of Birkeland and Eyde, the Badische Anilin and Soda Fabrik[11] uses long arcs with strong currents which are said to give excellent results.

Grau and Russ[12] have obtained a patent in the United States according to which the products of the reaction in the arc are walls, one would naturally sacrifice a slightly greater loss of heat. taken solely from the central part of the arc through cooled tubes. The electrodes are contained in a chamber into which the arc enters, while two or more tubes provided with cooling mantels project into the arc through which the products are sucked out. It is also possible to make one electrode hollow and draw the gases from the arc through this.

Besides the above, there should be specially mentioned the treatises of Muthmann & Hofer and of Brode. Muthmann & Hofer[13] have worked on the combustion of the atmoshperic nitrogen in the electric flame. For this purpose they used an alternating current of 2,000 to 4,000 volts and a current of 0.05 to 0.15 amp. Under these circumstances they obtained a flame of 0.4 to 3.5 cm. long under atmospheric pressure. In a flask of one liter capacity, they found after 15 to 20 minutes 3.7% nitric oxide. They correctly recognized that this was a case of equilibrium, though they were astray in regard to the temperature, and assumed the equilibrium to be purely thermal without proving it.

Brode[14] made his experiments with an electric flame of high voltage, and, besides deading with the question of equilibrium between nitric oxide and air, he especially investigated the reactions which take place in the different parts of the flame. In those experiments dealing with equilibrium, the air did not pass through the apparatus, but the flame burned in the four-liter bulb till samples of the gas showed a constant composition. With small flames this required a very long time, often an entire day. Using from 25 to 70 watts, and with Nernst filaments

[11] German Patent 40,300 (1905).
[12] United States Patents 884,919 and 884,920.
[13] Berichte der deut. chem. Gesellschaft, **36**, 438 (1903).
[14] Zeit. für Electrochemie, **11**, 752 (1905).

as electrodes, he obtained from 4.4 per cent. to 6.4 per cent. nitric oxide, the smallest percentage being obtained with the greatest watt consumption in large flames. With platinum electrodes he obtained 7.2 per cent. and with iridium, 5.8 per cent. nitric oxide. The latter result he ascribes to the decomposing influence of the fine particles of iridium on the nitric oxide in the flame. The unfavorable influence of the high watt consumption using the same arc length he does not explain. It was found in the course of the present investigation that the material of the electrodes exerted no influence, but that on the contrary the yields with platinum and iridium were in accord. It *was* found, however, that the watt consumption had a considerable influence.

All these processes and investigations rest on the assumption that it is expedient, in order to obtain the highest concentration, to bring the gases in the arc to a high temperature and then as quickly as possible to cool them so that they may quickly pass through that range of temperature in which a small nitric oxide concentration goes hand in hand with a great decomposition velocity.

Warburg and Leithauser[15] found that it was possible to obtain nitric oxide from the air by an electric discharge without the application of a high temperature. Their yields were small. Taking one example from their results, it was found that, with a current of 0.28 x 10-3 amp. and 9900 volts over the total circuit, the yield was 3 c.c. per hour at atmospheric pressure with a gas velocity of 13 c.c. per second. But these experiments are only important in that they disprove the conclusion of Berthelot, who stated as a result of his experiments that it was impossible to form nitric oxide in the electrical way without the application of heat.

Haber and Koenig[16] then attempted by means of the cooled arc to obtain results that would accordingly be due chiefly to electrical action. In the following investigation this is carried still further, but with an arrangement of apparatus that is quite different from that of Haber and Koenig. The experiments were carried on at first in a very high vacuum in which free electrons move very easily. The later experiments were performed under

[15] Annalen der Physik, **20**, 734.

[16] Zeitschrift für Electrochemie, **13**, 720 (1907).

pressures which gradually increased till about a pressure of one atmosphere was reached. In the later cases, a very short arc was used, while Haber and Koenig worked with very long ones, while still another difference lay in the fact that the later investigators used an alternating current, while in these experiments a direct current was used. By using as anode a hollow, water-cooled, silver capillary tube, by sucking the gases out through its centre, and with a hot cathode lying quite near the anode, it was possible to obtain either high percentages of nitric oxide or great yields per kilowatt hour by using appropriate pressure and gas velocity. The highest concentrations which had till now been obtained with air were between 9.5 per cent. and 10 per cent., which are those of Haber and Koenig at 100 mm. pressure. With the apparatus used in these experiments as high percentages were obtained, but with low voltage and at pressures between 300 and 400 mm. pressure. The yields obtained in the best commercial processes are given as 550 kilograms of nitric acid per kilowatt year, or 63 grams per kilowatt hour. In this investigation the yields obtained ran as high as 788 kilograms per kilowatt year, or about 90 grams per kilowatt hour. The most favorable conditions for these will be considered after the experiments themselves have been described.

THE CHARACTER OF THE DISCHARGE IN GASES.

Corresponding to the fact that in gases as in solutions ionisation is necessary for the passage of the current, it is possible to distinguish for gases three kinds of current according to the origin of the ionisation, viz., dependent, independent and mixed dependent currents.

If the ionisation is singly and solely the result of the action of an external ionizer, such as Roentgen rays, high temperature, etc., then the current is dependent. The current in this case exists then only so long as the ionizer acts, and the strength of the current depends only on the ionizer.

In an independent current the ionisation is called out by the impact of the moving ions themselves on neutral gas molecules, and cannot be obtained under a certain minimum electrode potential.

As the name indicates the mixed dependent current is the

result of the action of an external ionizer and of the impact of the ions.

The dependent current can be obtained in a gas by any difference of potential no matter how small. Beginning with the smallest potential, the current increases with increase of the potential, at first rapidly, then slowly, till it reaches an almost constant value—the saturation current. On still further increasing the potential, the current strength increases, *i. e.*, the velocity of the ions is increased till ionisation by impact is brought about,

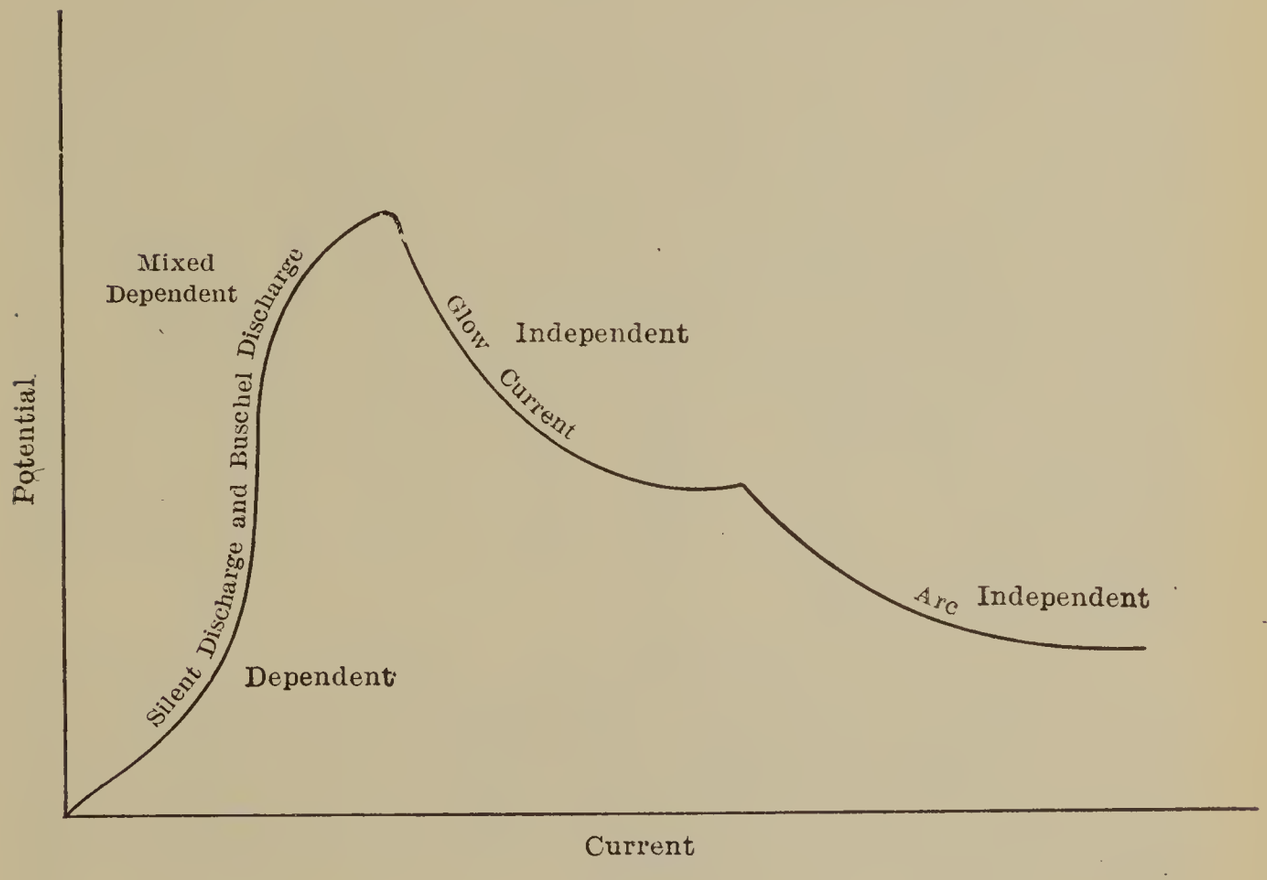


FIG. 1.

and this effect is added to that of the external ionizer. In general, at low pressures, this change of dependent to mixed dependent current occurs if the ion passes free through a potential fall of about 30 volts. This potential fall is necessary to impart to the ion an energy sufficiently great so that it can bring about ionisation of the neutral molecule by impact.

The accompanying curve or "characteristic," Fig. 1, shows the relation between these different currents as they appear on increasing the e. m. f. from zero, providing that a sufficiently

great resistance is in the circuit. If an external ionizer is used, for example Roentgen rays, it is seen at first as shown in the figure that the current increases with increasing potential till a maximum is reached, while the ionisation by impact of the ions brought about by still greater potential difference appears first near the electrodes which are accordingly surrounded by a mantle of light. The middle or intervening stretch of gas remains dark. The "characteristic" now shows an increase in the current due to the increased ionisation. As soon as ionisation takes place along the whole intervening stretch of gas the current increases further while the potential difference of the electrodes falls—we have then the glow current. By increasing the current now, more heat is generated, and if the additional resistance is not great enough, the heating of the electrodes, especially of the cathode, through the bombardment of the electrons, is so great that with the simultaneous increase in the current and decrease in the potential the electrical discharge is carried by the vapor of the electrode. Till recently such a discharge has been designated as an electric arc. The latter is nowadays no longer considered as identical with the discharge in the form of vapor of the electrode material, but is distinguished by the low value of the fall in potential at the cathode from the glow current, as in the case of the latter this potential fall is greater.

In the following investigation the discharge is a purely independent one in which the cathode fall is made relatively very small by using a hot iridium cathode covered with calcium oxide according to Wehnelt[17]. Vapor of the electrode material is not present in the arc. According to former nomenclature this would be designated as glow current, but is now spoken of as an arc.

Preliminary Experiments.

The preliminary experiments were made with an e. m. f. of 20 to 60 volts at pressures between 0.1 and 3.0 mm., the current strength being 0.1×10^{-3} to 0.2×10^{-3} amp. But the quantity of nitric oxide obtained at the lowest of these pressures was too small to be detected by the most delicate tests, and at 3 mm. pressure only traces could be found.

[17] Annalen der Physik, **14**, 425 (1904).

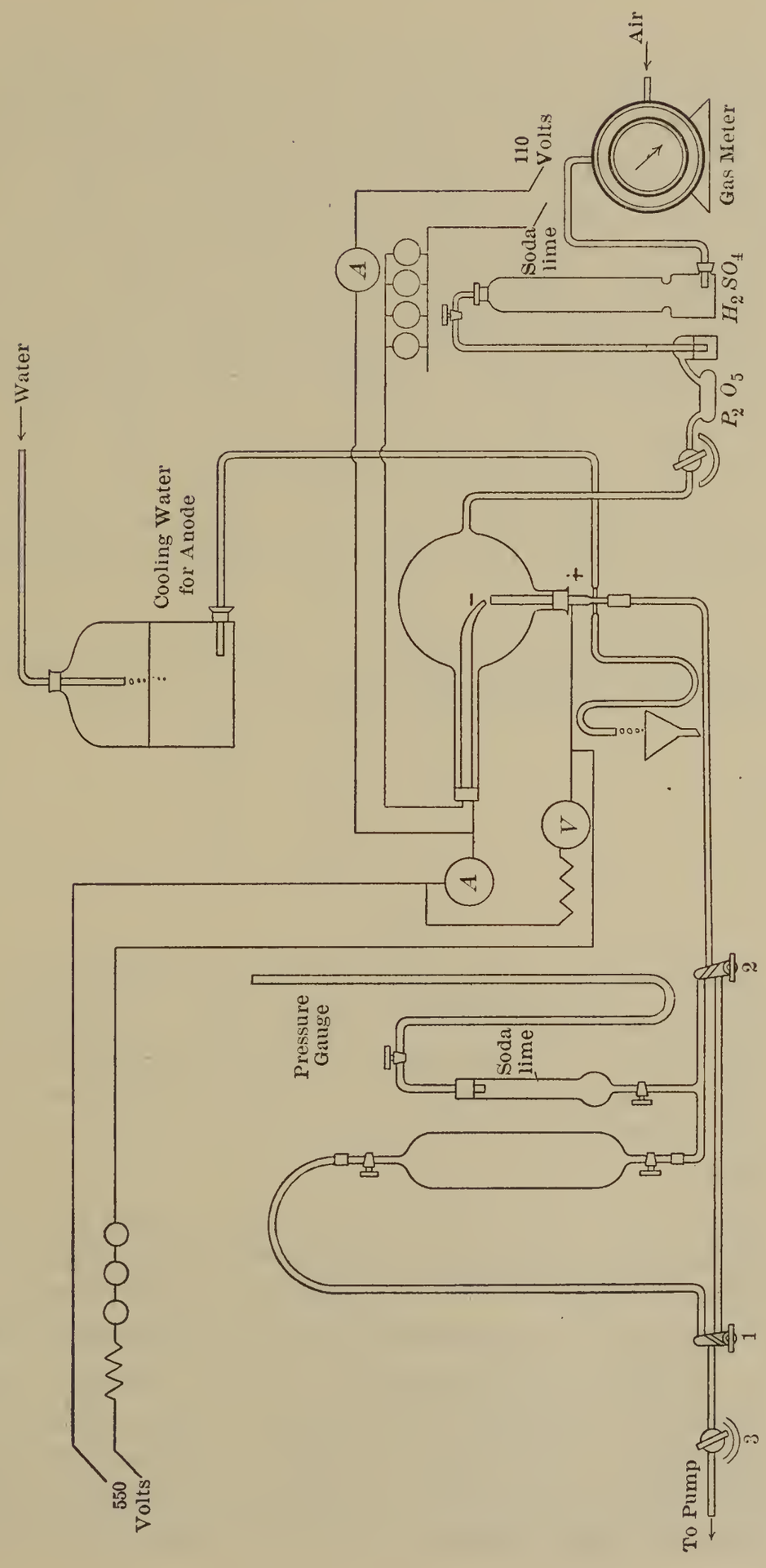

Air
Gas Meter
110 Volts
Soda lime
H_2SO_4
P_2O_5
Water
Cooling Water for Anode
A
V
Pressure Gauge
Soda lime
550 Volts
To Pump
1
2
3

FIG. 2.

Principal Experiments.

The experiments here described were carried out with pressures between 20 mm. and 700 mm. under the following conditions: Fig. 2 gives a diagrammatic representation of the apparatus. In Fig. 3, the arc chamber is shown on an enlarged scale. The air enters this bulb and is subjected to the action of the arc. Two copper rods, *a, a,* 2 mm. in diameter, pass through the glass stopper in which they are sealed air-tight by means of sealing wax. The end of each rod projecting in the bulb is about 4 mm. in diameter, and into this enlarged end a double strip of nickel, *b,* is held by screw. This nickel strip serves as the connection for the iridium cathode, the contact being only through spring pressure of the bent nickel pieces. Lying directly opposite the iridium cathode is the nickel anode, *d*,

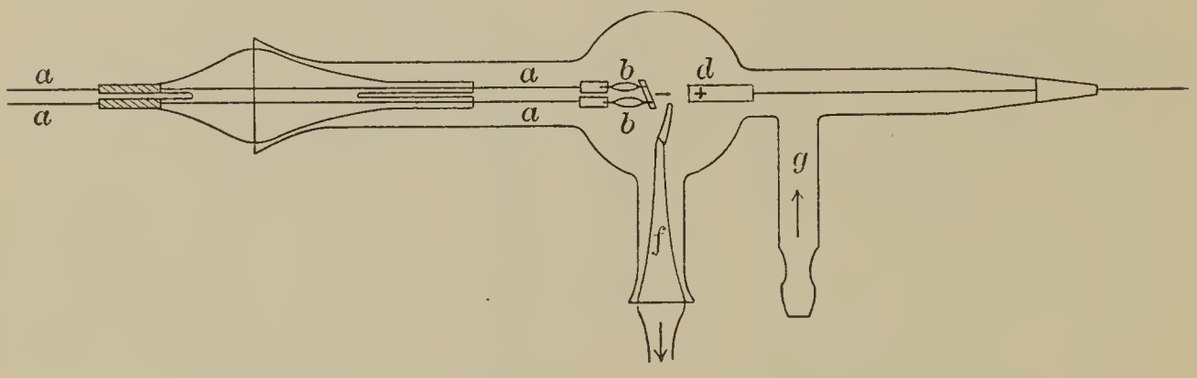


FIG. 3.

which is carried by a copper wire and is also sealed into its tube by sealing wax. From below, a glass tube, "*f*," projects into the bulb and bears on its upper end a hollow platinum cone, the upper end of which projects to near the lower side of the arc. This tube, "*f*," is movable in the outer tube by means of a ground glass joint.

In experiments 1-13 of Table I, the air entered the arc chamber through the tube, "*f*," and left through the tube, "*g*." In the experiments of Table II this arrangement was reversed so that the air was drawn out immediately after it had passed through the arc. In this last arrangement it would be more unlikely that air would be withdrawn that had not been subject to the action of the arc. In Table III are the experiments in which the nickel anode was pushed so close to the cathode that it made contact with the platinum cone on the top of the tube, "*f*,"

so that practically the air was withdrawn through the anode. In all the remaining experiments the arrangement shown in Fig. 4 was used. This anode is the silver, water-cooled capillary similar to that first used by Haber and Richardt, and now generally used in the laboratory of the Technische Institute, Karlsruhe, for the estimation of the equilibrium of gases in flames. This arc chamber shown in Fig. 2 is the principal part of the apparatus, the rest of the apparatus serving to measure, dry and collect the gas as well as to measure its velocity. The electrical part consists of the necessary measuring instruments and re-

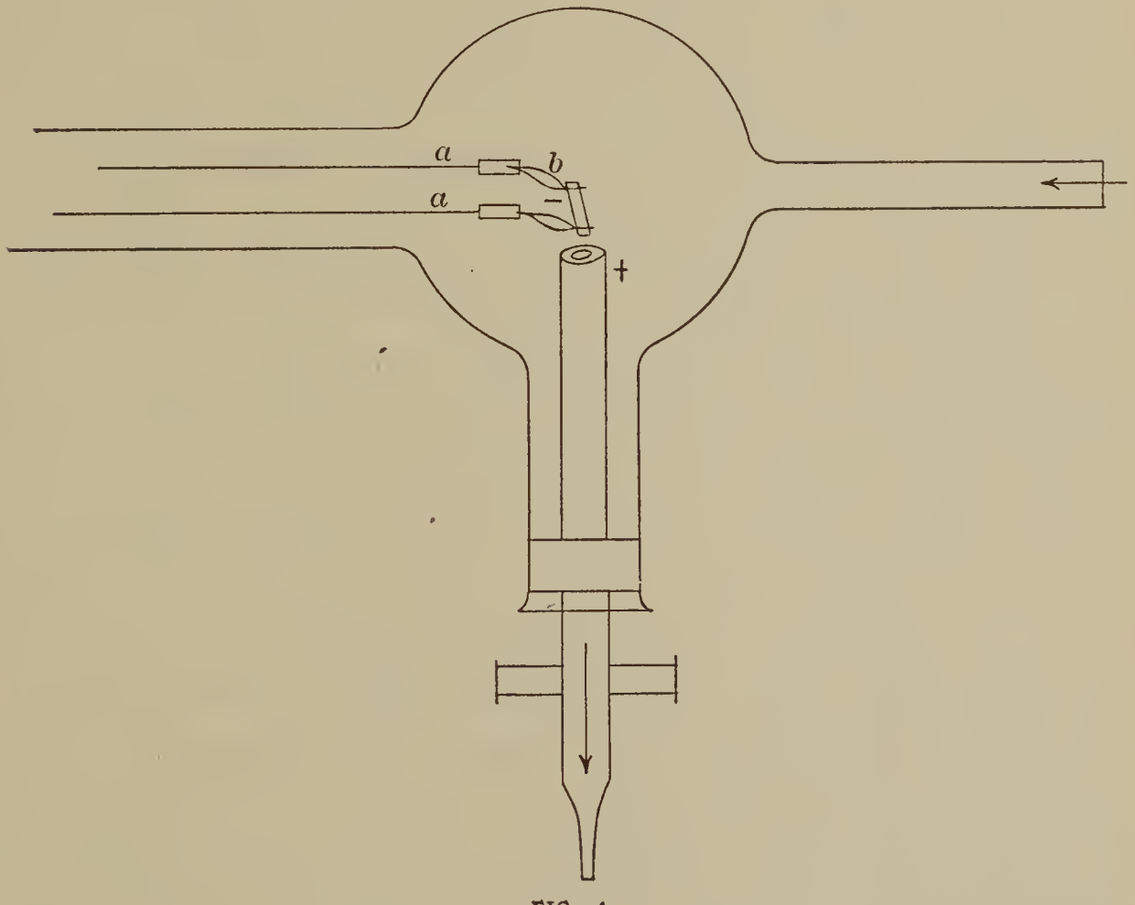


FIG. 4

sistances as shown in Fig. 2, as well as the generators not shown.

The air was drawn through the apparatus by means of a water suction pump. It passed first through a gasmeter so that it would be possible to measure the gas velocity, then through a soda-lime tower to absorb the carbon dioxide through sulphuric acid and phosphorus pentoxide for drying, then through a tap where the velocity could be carefully regulated, and then into

the arc chamber which it left by one of the above-mentioned methods. From this chamber the gas could either be led directly to the pumps, or through the gas pipette and then to the pump, according to the arrangement of the two two-way taps, 1 and 2, shown in the figure. In addition there was another tap for more careful regulation of the gas velocity, this tap beting between the two-way tap, 1, and the pump. The mercury manometer indicated the pressure of the gas at its entrance into the pipette which except at the greatest velocities was the same as in the arc chamber. This pressure gauge was protected against the action of the nitric oxide by means of a soda-lime tube.

By means of the two two-way taps it was possible to change the pipettes without interrupting the electrical current or that of the gas. By reversing these and by closing tap 3, the pressure in the arc chamber was kept constant. In experiments where the velocity was great the taps, 1, 2 and 3, were closed simultaneously. After closing the two taps at the end of the pipette the latter could be removed by disconnecting the rubber tubing at the ends. After inserting another pipette the tap, 1, was opened and the pipette evacuated to the required pressure, after with the taps, 2 and 3, were opened.

The copper conductors of the cathode were connected to the 110 volt circuit through a lamp rheostat and another variable resistance. The iridium cathode was 0.03 mm. thick and 1 to 1.2 mm. wide. The necessary heating current varied with the length and width of the iridium strip, varying between 4 and 7 amp. Experience soon taught one to recognize by the brilliancy of the iridium when it was near its melting point. Since the metal slowly disintegrated at the point where the arc originated and thereby became narrower, it was necessary, in order to prevent melting, to lessen the heating current during the progress of the experiments.

The current necessary for the arc was furnished by a one-half horse-power dynamo, which gave on open circuit about 550 volts at the electrodes. In the arc circuit were three 220-volt lamps and a slide rsistance of 220 ohms, all in series. The current strength varied between 0.1 and 0.25 amp. with a voltage at the arc varying in different experiments between 100 and 280 volts, corresponding to a power consumption of 20 to 50

watts. The arrangement of the electrical measuring instruments is shown in the figure.

In order to ignite the arc the apparatus was evacuated to a low pressure which varied according to the distance apart of the electrodes and to the temperature of the cathode. In general this pressure was between 100 and 200 mm., though with the shortest arcs it was easily obtained with pressures between 550 and 650 mm.

The water for cooling the anode was conducted through rubber tubing to the reservoir shown in the figure, from this through tubing to the anode, and finally in a similar manner to the waste pipe. The voltage being 550, the water column between the anode and the pipes was made as long as possible.

The iridium was moistened with a solution of calcium nitrate and heated strongly to obtain an adhesive layer of calcium oxide. The pipettes were of different sizes, approximating to 2,000, 500 and 150 c.c., according to the gas velocity, the smallest being used where the velocity was small, *i. e.*, between three and five c.c. per minute. After each experiment the pipette was washed out with conductivity water, then with alcohol, evacuated, strongly heated, and then cooled by leading carefully dried air through it. This treatment was required, since a trace of moisture would give too high percentage of nitric oxide.

Collecting and Testing the Gas.

The process by which an experiment was carried out was as follows: The cathode was heated and the apparatus evacuated directly through the two two-way taps till the arc was obtained. Then these taps were slowly reversed to bring the pipette in the circuit. The time was taken as well as the room temperature and the atmospheric pressure, the reading of the gasmeter recorded and those of the electrical instruments, these latter being adjusted if necessary by regulating the resistances.

Before collecting a sample of the gas, sufficient was allowed to pass through to amount to four or five times the volume of the apparatus so as to be sure that the original air was all swept out. At the end of the experiment the readings were again taken and the duration of the experiment determined. After removing the pipette, one of its ends was immersed in conductivity

water and the tap at this end opened till 75 to 100 c.c. water had entered. In order to completely absorb the gas in the pipette, it was shaken on a machine for at least two hours, and the solution obtained was titrated with barium hydrate solution after the addition of alcoholic solution of phenolphthalein. As soon as the pink color appeared, the pipette was washed out with pure water three times and again titrated.

For all the experiments in Table I, all in Table II with the exception of the last two, and for experiments 116, 117, 118 and 119 of Table IX, the barium hydrate solution was 0.054 normal. All the other experiments were titrated with 0.0838 normal solution.

In order to save time and to carry out a series of experiments rapidly, a number of pipettes were used. In the tables the numbers of these pipettes are given in each experiment and the following table gives the numbers and the volumes:

1.—2,197 c.c.	6.—2,423 c.c.	11.—154.6 c.c.	16.—458.4 c.c.
2.—2,350 "	7.— 562.4 "	12.—131.3 "	17.—439.0 "
3.—2,040 "	8.— 613.7 "	13.—551.6 "	18.—530.6 "
4.—2,870 "	9.— 564.0 "	14.—556.7 "	19.—541.5 "
5.—2,311 "	10.— 147.0 "	15.—566.7 "	20.—544.0 "

CALCULATION OF THE RESULTS.

From the observed values of the temperature and pressure and the known volume of the pipette, the volume of the gas in the latter at 0 and 760 mm. was calculated. The number of c.c. of barium hydrate solution multiplied by the normal factor of the solution gives the number of c.c. of nitric oxide and from this volume and the known volume of the pipette the percentage of nitric oxide is calculated.

In order to determine the relation between the quantity of nitric oxide obtained and the energy used, the readings of the ammeter and voltmeter are multiplied together to get the number of watts used in the arc.

The reading of the gas-meter was reduced to 0 and 760 mm., the barometer reading having been corrected for the vapor pressure of water at the room temperature. Having then determined the volume that passed through the apparatus, the volume per hour was calculated. When this is multiplied with the percentage of nitric oxide found, the number of c.c. nitric oxide per hour is obtained.

Table I.

No.	Press.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c.c.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric. Ac. gm per kw. hr.
									Amp.	Volts	Watts			
9	22	3	20	753	400	0.55	1.03	1.19	0.22	145	31.9	4.76	0.15	0.42
10	22	2	20	753	688	0.75	1.40	1.35	0.23	140	32.2	9.27	0.28	0.81
8	25	1	19	753	295	0.9	1.68	1.63	0.21	180	37.8	4.77	0.10	0.45
12	26	3	20	752	662	0.7	1.31	1.3	0.19	145	27.5	8.61	0.31	0.88
13	42	2	18	752	279	2.22	4.15	2.2	0.165	200	33.0	5.63	0.17	0.48
5	42	2	19	748	393	2.15	4.02	1.92	0.17	200	34.0	7.55	0.22	0.62
7	42	2	19	753	938	1.75	3.27	1.74	0.17	200	33.0	16.3	0.49	0.40
4	39	1	20	748	276	1.71	3.20	1.92	0.14	220	30.8	5.5	0.17	0.50
6	45	3	20	753	304	1.75	3.27	1.74	0.165	200	33.0	5.72	0.17	0.50
3	62	1	19	749	140	3.55	6.64	2.56	0.18	250	45.0	3.58	0.08	0.25
2	70	1	18.5	745	249	4.65	8.7	2.95	0.17	270	45.9	7.35	0.16	0.45
11	70	1	18	753	...	....	....	....	....	...	....	....	....	....
1	70	2	18	744	551	4.00	7.48	2.38	0.20	250	50.0	13.25	0.26	0.74

Table II.

15	50	2	17	756	396	2.9	3.5	2.41	0.19	160	30.4	9.55	0.31	0.89
16	60	2	17	755	567	4.2	5.08	2.9	0.17	180	30.6	16.45	0.53	11.50
17	64	2	18	753	620	4.3	5.2	2.8	0.165	190	31.3	17.4	0 55	1.56
18	70	4	18	753	560	6.55	7.93	2.85	0.16	190	30 4	15.96	0.52	1.47
19	72	5	19	753	581	5.2	6.3	3.1	0.165	190	31.3	17.5	0.56	1.57
20	74	2	19	753	698	5.93	7.17	3.4	0 17	200	34.	21.6	0.63	1.78
21	82	4	20	746	402	8.65	10.5	3.8	0.165	200	33.	15.5	0.47	1.32
22	100	5	18	746	390	9.62	11.62	4.1	0.165	200	33.	16.	0.48	1.36
23	...	...	...	...	...	...	...	...	...	...	...	...	...	...
24	190	6	20	748	520.	26.36	31.8	5.56	0.165	160	26.4	29.4	1.16	3.26
25	240	4	21	759	930	32.56	39 3	4.67	0.165	210	34.6	43.5	1.25	3.52
26	240	18	19	756	437	11.03	13.3	8.5	0.165	170	28.	37.2	1.33	3 74
27	240	16	20	756	521	8.8	10.65	7.94	0.185	200	37.	41.4	1.12	3 15
28	300	18	19	752	652	11.42	13.8	7.5	0.22	185	40.7	46.	1.13	3 15
29	...	...	...	...	...	...	..	...	...	...	...	...	...	...
30	300	16	21	745	431	6.3	11.78	7.5	0.22	185	40.7	30.2	0.74	2 80
31	300	18	21	745	791	7.22	13.3	6.97	0.22	125	27.5	55.3	2.00	5.62

Table III.

No.	Press.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c.c.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac. gm. per kw. hr.
									Amp.	Volts	Watts			
32	300	16	20	745	640	6.7	12.5	7.45	0.17	120	20.4	47.6	2.33	6 55
33	300	18	20	744	447	11.51	20.75	8.34	0.13	150	19.5	37.6	1.93	5.42
34	400	16	21	744	381	7.3	13.6	6.12	0.21	160	33 6	22.7	0.57	1.9
35	400	18	21	744	4350	8.0	15.0	5.8	0.25	140	35.0	252.0	7.2	20.2
36	400	16	21	743	573	7.5	14.0	6.29	0.19	170	32 3	36.0	1.12	3.15
37	400	18	21	743	218	8.35	15.6	6.05	0.20	170	34.0	12.3	0.35	1.02
38	600	18	21	752	412	12 83	23.9	6.33	0.11	130	14.3	26.1	2.6	7.3
39	590	18	21	748	368	12.22	22.82	6.06	0.11	160	17.6	22.2	1.28	3.6

Table IV.

132	100	10	23	747	542	0.45	0 84	4.74	0 22	110	24.2	25.7	1.06	2.98
131	100	11	24	747	994	0.35	0.65	3.1	0.22	105	23.1	30.8	1.33	3 7
130	100	10	24	747	1072	0.4	0.75	4.0	0.2	105	21.0	42.9	2.4	6 75

Table V—Pressure, 200 mm.

No.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c.c.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac gm. per kw. hr.
								Amp.	Volts	Watts			
147	10	23	755	76	1.06	3.00	8.3	0.19	105	19.95	6.30	0.31	0.88
148	11	23	755	126	1.7	3.18	8.48	0.19	105	19.95	11.80	0.67	1.88
146	11	23	755	245	1.5	2 8	7.5	0.19	100	19 00	18.30	0.92	2.59
145	10	23	753	151.6	1.5	2.8	7.81	0.21	75	15.75	11.80	0.75	2.11
101	10	23	751	327	1.4	2.62	7.4	0.185	80	14.80	24.20	1.63	4 60
108	11	25	751	268	1.35	2.52	6.6	0.185	110	21.27	17.70	0.83	2.35
107	10	25	751	512	1.1	2.05	5.7	0.19	100	19.00	29.18	1.53	4.21
103	11	24	752	540	1.2	2.22	6.0	0.17	115	19.55	32.40	1.66	4 65
99	9	23	751	1030	4.7	8.79	6.34	0.20	115	23.00	65.30	2.84	8.00
98	7	23	751	1445	4.3	8.04	5.9	0.19	120	22.80	85.20	3.74	10.50
97	8	21	752	3678	4.3	8.04	5.4	0 17	120	20.40	198.60	9.75	27.40
102	7	24	752	31920	1.1	2.05	1.52	0.17	135	22.95	485.10	21.80	61.30

Table VI—Pressure, 300 mm.

96	10	20	752	239	2.8	5.23	9.9	0.185	90	16.65	23.66	1.42	4.00
142	10	24	753	298	2.62	4.86	9.15	0.18	120	21.60	27.11	1.22	3.41
143	11	24	753	298	2.70	5.05	9.7	0.18	119	19.80	28.90	1.46	4.10
144	11	24	753	298	2.70	5.05	9.7	0.185	115	21.20	28.90	1.37	3.85
88	8	17	756	642	10.92	20.38	9.0	0.185	135	24.90	57.78	2.55	7.17
94	7	18	752	1195	8 73	16.27	7.85	0.19	90	17.00	93.20	5.46	15.35
93	8	19	750	2080	8.04	14.96	6.63	0.185	105	19.42	137.96	7.10	20.00
86	16	19	761	5350	5.70	10.66	6.28	0.185	110	20.55	335.98	16.60	46.60
85	17	17	761	71000	4.50	8.41	5.20	0.165	110	18.15	396 20	20.20	56.70
84	8	17	761	9000	4.10	7.66	3.38	0.19	104	19 95	304.20	15.20	42.70
83	9	17	761	12960	3.02	5.61	2.68	0.19	110	20.90	347.30	16.60	46.60
82	7	17	761	23400	2.21	4.11	1.98	0.17	115	19.55	403.20	23.70	66.66

TABLE VII—Pressure, 400 mm.

No.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c c.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac. gm. per kw. hr.
								Amp.	Volts	Watts			
95	11	19	752	195	3.85	7.2	9.5	0.165	95	15.67	18.5	1.15	3.24
91	11	19	751	360	3.8	7.1	9.4	0.185	110	20.35	33.8	1.44	4.05
92	10	19	751	389	3.5	6.54	9.1	0.185	120	22.2	35.3	1.59	4.47
81	8	17	757	532	14.1	26.36	8.73	0.165	100	16.50	46 4	2.97	8.35
89	10	17	757	563	3.3	6.17	8.5	0.165	150	24.75	47.8	1.93	5.42
80	7	17	757	1470	11.2	20.94	7.56	0.19	95	18.05	111.1	6.15	17.30
77	7	17	750	2032	10.7	20.00	7.22	0.155	120	18.60	146.8	7.85	22.10
74	9	16	752	5350	7.4	13.84	4.95	0.165	110	18.15	264.8	14.60	41.00
73	7	17	752	10500	5.8	10.84	3.9	0.185	110	20.35	409.5	20.00	56 20
71	7	17	752	17280	4.2	7.85	2.83	0.14	140	19.60	489.0	25 20	70.80
70	8	17	752	20580	3.5	6.54	2.2	0.14	140	19.60	452.7	23.10	65.00
106	7	24	751	30600	3.1	5 80	1.7	0.21	120	25.20	520.0	20.70	58.20
69	9	17	752	38400	2.3	4.30	1.54	0.115	200	23.00	591.30	25.70	72.20
65	8	21	749	56820	2.0	3.74	1.25	0.17	170	28.90	710.2	24.50	69.00
105	9	23	751	60300	2.15	4.02	1.18	0.11	250	27.50	711.4	25.80	72.60

TABLE VIII—Pressure, 500 mm.

No.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c.c.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac. gm. per kw. hr.
								Amp.	Volts	Watts			
68	12	23	744	340	4.1	7.56	9.66	0.15	120	18.00	32.8	1.8	5.06
67	12	23	744	352	3.9	7.30	9.17	0.15	95	14.25	32.27	2.26	6.35
66	12	23	749	287	3.2	5.98	7.50	0.185	95	17.57	21.5	1.20	3.37
63	9	22	755	717	14.5	27.10	8.02	0.155	115	17.82	57.5	3.23	8.80
62	7	21	755	1218	14.3	26.50	7.76	0.155	115	17 82	94.5	5.30	14.90
61	8	22	758	2020	13.8	25 80	6.93	0.165	120	19.80	140.0	8.02	22.60
59	9	21	758	6840	8.3	15 52	4.09	0.165	110	18.15	279 7	15.30	43.00
60	7	21	758	16820	4.7	8 79	2.57	0.155	120	18.60	427.1	23 00	63.70
55	9	20	762	18600	4.6	8.60	2.50	0.13	135	17.55	465.0	26.50	73.20
54	8	20	762	24900	4.4	8.23	2.20	0.13	150	19.50	547.80	28.00	78.70
58	8	22	762	24720	4.3	8.04	2.18	0.155	165	25.37	538.9	21.10	59.20
53	9	19	759	35760	4.0	7.48	2.16	0.165	175	28.87	773.4	26.70	72.20
104	8	22	751	48480	3.15	5.29	1.60	0.14	180	25.20	775.7	30.80	86.60
52	7	19	759	59280	2.8	5.23	1.52	0.13	210	27.30	901.0	33.00	92.70
57	9	21	762	57300	2.8	5.23	1.52	0.15	190	28.50	870 9	30.60	86.10
113	9	25	744	59400	2.9	5·42	1.60	0.115	260	29.00	950.4	32.80	92.20
149	13	24	757	59100	2.3	4.30	1.29	0.09	280	25.20	765.0	31.50	88.50
150	14	24	757	62000	2.8	5.23	1.58	0.12	240	28.80	967.0	32.37	91.10
151	15	23	757	62000	2.7	4.05	1.47	0.115	255	29.30	911.3	31.10	87.40
157	8	23	757	63878	2.7	4.05	1.36	0.11	260	28.60	868.8	30.55	85.80

TABLE IX—Pressure, 590 mm.

No.	Pipette	Temp.	Barom.	Veloc. c.c. per hr.	Bar. Hyd. c.c.	Nitric Ox. c.c.	Nitric Ox %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac. gm. per kw. hr.
								Amp.	Volts	Watts			
56	7	21	762	344	17.96	33.5	8.21	0.14	120	16.8	28.24	1.7	4.77
51	8	19	759	447	17.57	32.82	7.51	0.12	140	16.8	33.56	2.0	5.62
47	7	19	752	558	15.74	29.4	7.22	0.12	140	16.8	40.28	2.4	7.40
46	9	18	752	1060	14.63	27.3	6.6	0.115	130	14.95	70.00	4.68	13.10
45	8	23	752	2410	11.62	21.78	4.87	0.165	195	32.17	167.30	5.21	14.65
101	10	23	751	24780	1.44	2.62	2.99	0.155	190	29.45	740.90	25.50	71.60
109	8	25	752	30720	5.91	11.05	2.53	0.155	190	29.45	777 20	25.80	72.50
49	9	20	752	36780	3.82	7.15	1.75	0.1	200	20.00	643.60	32.30	90.00
50	11	17	759	60730	2.2	4.12	1.3	0.115	220	25.30	789.30	33.20	92.80
114	7	24	744	60480	3.35	6 25	5.56	0.165	205	33.82	943.50	27.90	78.50
115	8	24	744	63420	3.5	6.55	1.5	0.165	205	33.82	951.30	28.10	79.00
116	18	24	744	64500	4.5	5.45	1.46	0.17	195	33.50	945.00	28.20	79.20
117	16	24	744	64500	4.17	5.07	1.54	0.165	215	35.47	993,00	28.00	79.00
118	17	24	744	71400	3 53	4.27	1.38	0.155	230	35.00	985.30	28.20	79 20
119	9	24	744	70980	4.79	8.85	1 44	0.15	240	36.00	1022.00	28.30	79.60
120	13	24	745	69400	2.8	5.23	1.33	0 15	250	37.50	923.30	24.30	68.28
152	19	23	757	49500	3.55	6.63	1.71	0.115	250	28.75	846.50	29.40	82.60
153	15	23	757	54600	3.3	6.16	1.51	0.09	280	25.20	824.30	32.70	91.89
154	20	23	757	43750	3 92	7.33	2.1	0.115	240	27.60	918.70	33.20	93.30
155	9	23	757	45280	4.00	7.47	1.85	0.11	250	27.50	837 70	30 46	85.60
156	7	23	757	54600	3.50	6.55	1.63	0.10	265	26.50	889.90	33 60	94.40
With Iron Cathode.													
138	9	19	751	11496	9.4		4.3	0 30	80	24.00	494.30	21.70	61.00
139	8	19	751	17520	10.5		4 41	0.29	105	30.40	772.60	25.40	71.50
140	7	19	751	24300	8.9		4.08	0.27	140	37.80	991.40	26.20	73 60
133	7	24	750	8760									
134	9	24	750	49500	None		None				None	None	None
135	8	24	750	31980									
136	16	24	750	354000									

TABLE X—Pressure, over 700 m.

No.	Press.	Pipette	Temp.	Barom.	Bar. Hyd. c.c.	Veloc. c.c. per hr.	Nitric Ox. c.c.	Nitric Ox. %	Current			Nitric Ox. c.c. per hr.	Nitric Ox. lit. per kw. hr.	Nitric Ac. gm. per kw. hr.
									Amp.	Volts	Watts			
129	707	7	23	751	3.9	60000	7.29	1.51	0.17	195	33.15	906.0	27 3	76.7
121	705	8	22	750	3.55	59460	6.63	1.26	0.155	190	29 40	688.8	23.5	66.0
122	710	7	22	750	2.95	57900	5.51	1.13	0.17	170	28.90	654.2	22.7	63.7
123	710	8	22	750	3.2	56840	5.98	1.13	0.17	172	29.24	642.0	21.9	61.6
124	710	9	22	750	3.0	56820	5.61	1.16	0.17	172	29.24	659.0	22.5	63.2
127	725	8	24	751	4.5	55500	8.41	1.63	0.155	220	34.10	904.0	26.6	74.8
126	705	7	23	751	4.0	51600	7.48	1.56	0.185	170	31.40	804.0	25.6	72.0
128	709	9	23	751	3.8	47400	7.10	1.44	0.17	210	35.70	682.5	19.1	53.5
125	723	9	22	751	7.05	33560	13.18	2.60	0.13	270	35.10	875.1	24.9	70.0

TABLE XI—Pressure, 590 mm.

							(liters)							
158			24	757	49.1	80760	9.18	1.24	0.12	260	31.20	1001.5	32.1	90.6
159			24	757	62.7	57390	11.72	1.58	0.12	240	28.80	907.2	31.5	88.6
160			24	757	44.8	48440	8.38	1.60	0.11	250	27.50	775.0	28.2	79.2

THE RESULTS OF THE EXPERIMENTS.

Table I gives the results of 13 experiments which were carried out with pressures between 22 and 70 mm. and with velocities which were from 7 to 17 c.c. per minute. The platinum glowed dark red. The length of the arc was about 17 mm. The cathode was dark red with the exception of the very small place where the arc rested which was white hot. This point did not remain constant but wandered over the surface of the cathode. The arc had an elliptical form and a pink violet color which at the surface of the cathode was a lavender blue. From the table it is seen that the per cent. varies between 1.19 and 2.95. It is plain that this concentration increases with the pressure and falls with the velocity. The influence of velocity is seen by a comparison of experiments 1 and 2 which were carried out at the same pressure of 70 mm. A similar relation exists between those experiments which were carried out at 42 mm., but it is seen that, although the per cent. of nitric oxide is smaller, the yield per kilowatt hour is greater, *i. e.;* it increases with the velocity.

In order to determine the influence that the heating of the cathode exerted on the result, experiments 5, 7 and 8 were performed without any heating current, so that the cathode after ignition of the arc was only heated by the arc itself. No difference is seen between these experiments and those carried out where the other circumstances were similar. Experiment 1 was carried out without any arc at 70 mm. while the cathode was heated to the highest possible temperature without melting. The gas was drawn through very slowly, but not a trace of nitric oxide was found in the pipette.

Since it was possible that a part of the gas leaving the platinum cone might be taken from the arc chamber without coming into the arc, the arrangement was reversed so that the gas left the arc chamber through the platinum cone and the platinum was pushed as nearly as possible to the arc. Table II gives the result of a series of 14 experiments carried out in this manner. The first four gave a little better result than the preceding series, and accordingly the same arrangement was retained for those at higher pressure, which also gave still better results. In ex-

periments 25, 28, 30 and 31 no heating current was used, and the percentage was not adversely affected. Since Brode asserted that platinum gave better results that iridium, experiments 26 and 31 of this series as well as some others of the following series were made with platinum as cathode, but the results obtained gave no confirmation of Brode's statement. In order to carry out the experiments at higher pressure without too great an increase in the voltage, the distance between the electrodes was reduced to 6 mm. In experiment 26 this distance was only 4 mm.

In order to obtain this shorter arc for the higher pressures the anode was shoved in till it came in contact with the platinum and the arc rested more or less completely on it.

The next series shown in Table III comprises experiments 32 to 39 with pressures between 300 and 600 mm. It was quite difficult to prevent the platinum cone from melting and this gradual melting of its edge so shortened the platinum anode that the gas was not taken out of the arc.. No heating current was used in this series with the exception of 37 and 36. Evidently in experiment 39 there is a mistake as the arc was only 2 mm. long and many subsequent experiments showed that it was impossible to obtain so high voltage under such circumstances. From experiment 35 we see how greatly the yield increases with the gas velocity.

In order to retain the advantage of a short arc as well as to prevent the heating of the anode, which was especially great with increased velocities, recourse was had to a cooled anode—the water-cooled capillary of Haber and Richardt. The length of the arc could be easily regulated by bending the copper conductors of the cathode. The arrangement is shown in Fig. III, B. There are two points which had to be principally kept in view. First, the maximum concentration at the different pressures was to be found, and, second, to determine the best yield per kilowatt hour. For the first purpose, small velocities were required; for the second, great velocities; while low pressures (up to a certain limit) proved more profitable for high concentration, and high pressures gave better yields per kilowatt hour. As mentioned, this holds within certain limits, for, while greater concentrations were obtained at 300 mm. pressure with small velocity than at 400 and 500 under like circumstances a lowering

of the pressure to 200 and 100 mm. did not give still higher concentrations.

A few experiments were made to see the general trend of the results, and it was seen that at 100 mm. pressure poorer concentration and yields were obtained than at higher pressures. Table IV gives the results of three such experiments, and it is seen that only 4.75 per cent. was reached. In order to prevent as far as possible mistakes in titration at lower pressures, it would be necessary to work with pipettes of great volume, and so no experiments with small velocity were made. In order to pass through the required quantity of air the duration of the experiment would be unduly long.

Table V contains the results of 12 experiments with gas velocities between 1.5 and 590 c.c. per minute. The percentage of nitric oxide lies between 1.5 and 8.5 per cent., while the yield of nitric acid per kilowatt hour has increased to 61.3 gm. In 97 the cathode was of platinum and in 101 there was no heating current without in either case there being any noticeable change in the results.

In Table VI are found the experiments at 300 mm. pressure. Here the concentration reaches almost 10 per cent. and the yield of nitric acid 66 gms. per killowatt hour. The length of the arc varied between 2.5 and 3 mm. In experiment 85 the high yield is to be ascribed to the small current used. This small current was due to a slightly longer arc and the use of a higher resistance by which means the voltage at the arc was kept low. Two experiments were here carried out with a platinum cathode. Experiments 142, 143 and 144 were made to confirm the good result found in 96. In 144 no heating current was used.

Table VII gives the results at 400 mm. It is seen that the high concentration obtained at 300 mm. is not reached, but the yield of nitric acid per kilowatt hour is better. The length of the arc was between 2 and 3 mm.

In Table VIII the yields of acid per kilowatt hour with greater velocities are still better. Experiments 149, 150, 151 and 157 were performed to confirm the results of 52 and 113, which it seems were due to the small current used. The results show that within certain limits the short arc with strong current does not give as good a result as a longer arc. If, however, the gas

velocity becomes too great the yield falls again, and in addition the arc is so unstable that it is quite easily extinguished. It is evident that with the high velocities it is impossible to do without the heating current. With the passage of 500 c.c. to 1,000 c.c. of gas per minute through the opening of the anode, which was about one mm. wide, it would be impossible to prevent the cooling of the adjacent cathode if there was no heating current to such an extent that the arc would be extinguished. Experiment 66 was the single one which was carried out without a heating current, but the slight concentration obtained is not due to this fact, but to the fact that not enough gas was sent through the apparatus to remove the original air.

Table IX comprises 21 experiments in which the high yields at high velocities are shown. The reason for taking a pressure of 590 mm. that the manometer was only long enough to show readings slightly above 500 mm. Experiments 114-120 constitute a series which were carried out in rapid succession, under the same conditions, to determine if as good yields as those shown in 49 and 50 could be again obtained. The results were uniformly smaller. The reason for this is that with this gas velocity a small watt consumption would produce as great a quantity of nitric oxide. In order to establish this for a certainty, experiments 152-156 with a smaller watt consumption were made and with the favorable results as shown. Experiments 45-47 were made with a platinum cathode.

Experiments 138-140 were performed with an iron cathode and represent the result of two and one-half days' work. The iron electrode—an ordinary short nail—was oxidized at its end in an oxygen flame. It was then fixed in the end of one of the copper conductors, but no heating current could be used. In order to make the arc the nail was brought as close as possible to the anode, while between the electrodes there was a potential difference of 600 volts. A vacuum of 16-20 mm. was required to start the arc, which as it burned became longer till it was extinguished. Various methods were tried to start the arc but that which was used for the three successful experiments consisted in winding a short piece of fine wire around the nail and allowing the wire to touch the anode. This wire would eventually melt as the pressure increased till finally the arc burned between

the nail and the anode. By a comparison of experiments 140 and 110 it is seen that the yields with the same gas velocity is about the same, though the percentage is appreciably higher with the iron.

Another manometer was used for the experiments in Table X. With a water suction pump, at these higher pressures, a velocity greater than a liter per minute could not be obtained. In experiments 122-124 the cathode was a Nernst filament through which a platinum wire had been drawn. The Nernst filament was moistened with a few drops of calcium nitrate. Number 128 was performed without any heating current, but a higher voltage was necessary to prevent the extinguishing of the arc.

Since with pipettes of 500 c.c. capacity the product of the action of about a half a minute only was obtained, requiring 3 or 4 c.c. of the barium hydrate solution, it will be seen that a slight titration error might give results considerably too high. It was accordingly considered advisable to lead the gas through a glass spiral that was cooled with liquid air so as to condense the product obtained in from five to ten minutes. In order to give the nitric oxide a sufficient time to be oxidized before it entered the cooling tube, a pipette of two liters capacity was inserted between the arc chamber and the spiral. There must then be sufficient NO_2 present to ensure the formation of N_2O_3 since any unchanged NO would be lost on account of the vapor pressure of the NO being too great to permit its being completely liquified at the temperature of liquid air.

Fig. 5 shows the arrangement of the apparatus for these special experiments: "A" is the mixing chamber, "B" is the cooling spiral in the liquid air vessel, and "C" is a pipette of 500 c.c. capacity which was inserted between the liquifier and the pump. This latter was for the purpose of determining if any solid N_2O_3 was blown through the apparatus, or if any NO had not been oxidized. In the perpendicular glass tube that formed one end of the cooling spiral, glass wool was inserted for a length of about 10 cm. in order to catch any traces of N_2O_3 in the form of dust or cloud. The arc was ignited at low pressure, the taps so arranged that the air passed through the apparatus at gradually increasing pressure directly to the pump without passing through the condenser. Having obtained the required ve-

locity of a liter per minute, the condenser was slowly lowered into the liquid air, the time required being about five seconds. At the moment that the spiral was immersed in the liquid air. the time, the readings of the ammeter and voltmeter, as well as that of the gas meter were taken. In about five minutes the taps of the liquifier were closed and the pipette "C" closed while the time and the readings of the instruments were again taken. In order to determine the yield per kilowatt hour it was only necessary to take the reading of the electrical instruments and know the duration of the experiment. In order to find the per cent. nitric oxide, the quantity of air that enters must also be obtained from the gas meter. On removing the spiral from the liquid air

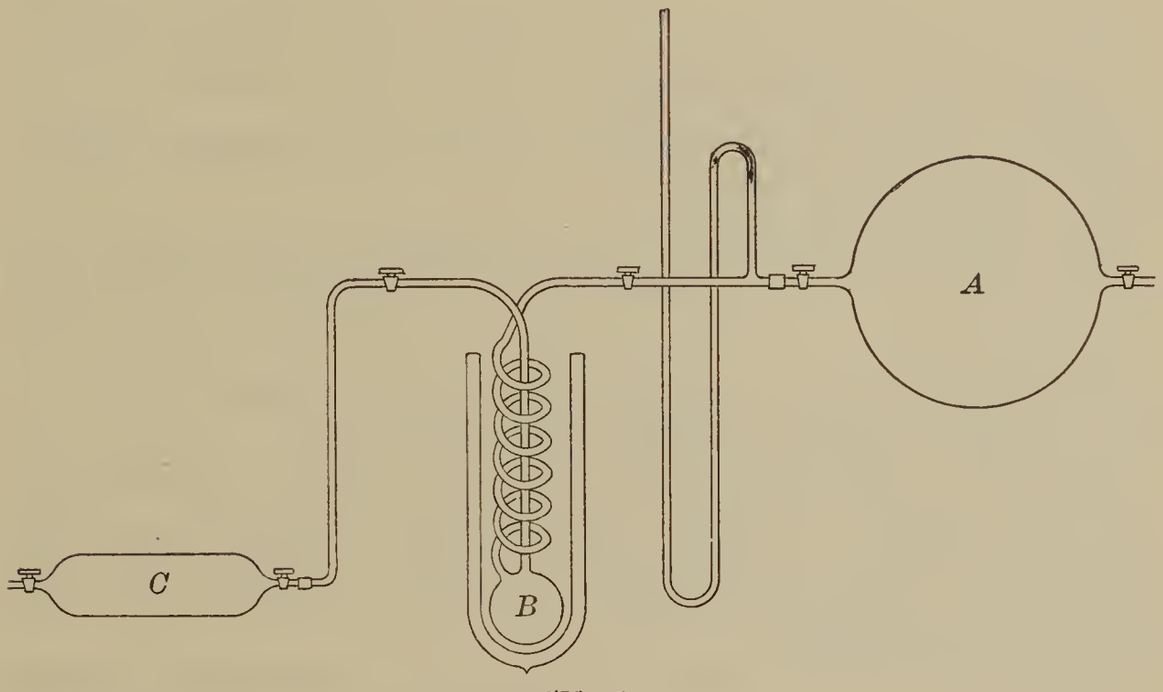


FIG. 5.

some bright blue solid N_2O_3 was seen in the glass wool as well as in the upper turns of the spiral. In order to determine the nitric oxide the cooling spiral was disconnected at the rubber connection and one end connected with a pipette that had been evacuated with the water pump but not dried. The tap between the glass spiral and the pipette was opened so that when the N_2O_3 had melted and vaporized, it was mostly driven into the pipette. In order to remove the last traces of the gas from the liquifier the free end of the latter was connected with a drying tower containing sulphuric acid and a soda-lime tube. On opening the tap between the condenser and the drying apparatus the

air entered and swept the last traces of gas into the pipette. That end of the still partially evacuated pipette that had been connected with the condenser was dipped in water and the tap opened till about 75 to 100 c.c. of water had entered. After shaking for a couple of hours on the machine the solution was titrated.

The results shown in Table XI are in such good accord with those obtained by direct collecting of the gas that there can be no doubt as to their accuracy. A comparison with the results of Table IX shows that the results are almost the same for the same velocities.

RESULTS.

The following conclusions can be drawn from the experiments as given in the preceding tables:

Higher concentrations of nitric oxide were obtained by taking the gas directly from the arc.

The highest concentrations were obtained at pressures between 300 and 500 mm. by sucking the gas out of the arc through a cooled anode.

Small gas velocities are favorable to high concentrations, and great velocities to high yields per killowatt hour.

For every gas velocity there is a maximum concentration possible so that an increase of the current strength above that necessary for this concentration results only in a decrease in the yield per kilowatt hour.

The best yields per kilowatt hour are obtainable at high pressures.

The highest cncentration obtained was 9.7 per cent. and the highest yield was 92 mg. per kilowatt hour.

University of Toronto,
October, 1908.

DISCUSSION.

MR. ALLERTON L. CUSHMAN: Was there any variation in the moisture content of the gases that were passed through the arc? I understand that they have arrangements for determining this,

and I believe it was observed that the amount of moisture content in gases affected the results in such experiments.

MR. MORDEN: No changes were made; the experiments were all made under the same conditions, with dry air, and no experiments were made to see what would be the effect if moisture were introduced.

MR. MORDEN: No changes were made; the experiments were influence of the portion of the arc upon the composition of the gas, that is, the portion of the arc from which the gases were removed? Did it vary, and if so, how much? Was there any sort of curve drawn showing the composition of the gases, as removed from different portions of the arc, under otherwise the same conditions?

MR. MORDEN: No. The arcs were from three to six millimeters, probably four millimeter lengths were the average.

MR. LIDBURY: Under those circumstances it would be rather difficult. In the case of arcs of any considerable length I think very interesting results would be obtained. Such measurements in the case of spark discharges in gas at low pressure give some very interesting results.

MR. MORDEN: Muthmann & Hofer and Brode carried out some experiments of that kind, and Brode has given names to the three different parts of the arc: One the zone in which nitric oxide is formed, another in which there is a decomposition of the ozone and the other in which decomposition of the nitric oxide takes place. These are flaming arcs, and it is possible in these to determine the composition of the different parts, but you cannot do it with these short arcs.

MR. L. F. GUTTMAN: Have you noticed any effects on the rubber connections, and is there any effect on the rubber which would be lost at larger velocities?

MR. MORDEN: A rubber tube having been used for six months showed no indication of any action. We brought the ends of the glass tubes in contact, and this, of course, exposed only a very small surface of the connecting rubber tube.

A paper read at the Fourteenth General Meeting of the American Electrochemical Society in New York City, October 30, 1908; President E. G. Acheson in the Chair.

THE THEORY OF ELECTROLYTIC PAINTS.

By Wilder D. Bancroft.

If one reads the articles that have been written on the electrolytic production of paints and especially on the electrolytic production of white lead, one is struck by the fact that most of the time has been spent in trying to make a product which will have the proper chemical composition while there is very little which bears directly on the covering quality of the paint or on the amount of oil which it absorbs. Since white lead is a definite chemical compound,[1] the problem of making a white lead of the proper composition seems to a man who has never done it about as foolish a matter as wrestling with the problem of how to make sodium chloride or copper sulphate of constant composition. So far as I can see, anybody can do it. Even if it were difficult, which I don't admit, it is not very important because the real problem is to make paint and not chemical compounds. The so-called physical properties of white lead or of other paints are the important things and those are the points in which the electrolytic pigments are unsatisfactory. As far as that goes, our whole knowledge of the subject is meagre, to say the least. White lead, made by the old Dutch process, is apparently the standard; but I have never been able to find any satisfactory statement why this process gives the best paint. While I am not prepared to say that the electrolytic manufacture of white lead or other pigments will ever be a commercial success, I am certain that the first need is to study how the so-called physical properties vary with the conditions of the formation.

In a small way, we have been trying to study this problem recently at Cornell. We have not got very far as yet, and I

[1] Hawley, Jour. Phys. Chem., **10**, 654 (1906).

have not much in the way of results to lay before you; but it seemed to me worth while to call your attention to this important field because it is one that has been neglected as far as published results are concerned.

So far we have considered only the size of the crystal in our work, and we have studied the chemical precipitation of lead chromate and the electrolytic precipitation of so-called cuprous oxide. The work on the lead chromate was done a few years ago by Mr. E. E. Free, and that on the cuprous oxide last spring by Mr. D. Miller. The paragraphs in quotation marks refer to Mr. Free's paper, which will probably be published in the February number of the Journal of Physical Chemistry.

"A molecular solution of $Pb(NO_3)_2$ was prepared by dissolving 331 grams of the crystalline salt in a liter of distilled water; 500 cc of this solution were than diluted to one liter, 500 cc of the resultant solution again diluted to one liter, and this process repeated until a series of fifteen solutions was obtained, each member of which had a concentration equal to one-half that of the preceding member. A similar series of solutions of K_2CrO_4 was then prepared, starting with a molecular solution (194.5 grams per liter). Thus the solutions of corresponding number in the two series were chemically equivalent, the most concentrated being molecular, and the most dilute M/16384.

"Preliminary tests showed that the manner of mixing the solutions in making a precipitation had much influence on the character of the precipitate produced—probably because of concentration changes occasioned by incomplete mixing. This trouble was partially avoided by the following method of precipitation: Fifty cc of one of the solutions were placed in a 200 c.c. beaker and stirred vigorously by means of an electrically operated stirrer, while 50 cc of the equivalent solution of the other series were added in a slow stream. The mixture was then stirred for exactly five minutes, then taken out and the character of the precipitate examined. The same 50 cc pipette was used in all cases to measure the solutions.

"Accidental and irregular variations in the precipitate are not by any means entirely avoided by this method of precipitation, but the results are much more uniform than by the ordinary method of pouring the solutions together. For precipitations

at temperatures above that of the room, the beaker in which the precipitation was made was immersed in a dish containing water at the desired temperature. It seemed, on trial, that it made no constant difference in the character of the resulting precipitates whether the chromate solution was poured in the lead solution, or vice-versa; consequently the former order was adopted in all cases. The precipitates were examined by the eye for color and state of aggregation (coarseness), comparison being made at different concentrations. Sedimentation tests were made by diluting the precipitates with their mother-liquor until all contained the same quantity of precipitate in unit volume, and observing the rate at which these mixtures settled when placed in test tubes. All of these methods of comparison gave more or less erratic results, which were probably due in part to faults in the method, and in part to accidental variations in conditions. By far the most reliable indications were given by the microscope, and the best method of observation was found to be as follows: A small drop of the precipitate and mother liquor were placed on a slide, covered with a cover glass, and the latter rubbed round a little to break up the aggregates of the crystals. The most important parts of the slide were then observed with a magnification of 1100 diameters and the average size of the crystals determined by means of a micrometer eye-piece.

"The precipitates were subject to very great irregularities. Two precipitates prepared as nearly as possible in the same manner will differ materially in color, crystal size, tendency to flocculate, etc. These variations are undoubtedly due to uncontrolled differences in the manner of precipitation, although what these differences may be it is impossible to say from the knowledge at present available. It must be understood, of course, that all the variations in the precipitates are only relative. All the precipitates are yellow; all of them are composed of very fine particles. In fact, all have the characteristic properties of precipitated $PbCrO_4$. The differences appear only on close examination.

"It is believed that all the precipitates are crystalline, although in a few cases it was impossible to detect individual crystals. In most cases the precipitate appears under the microscope as a mass of interwoven needle-like prisms, which are very easily

broken up into short prismatic fragments. The crystal angles could not be determined. The smalled fragments almost invariably showed strong Brownian movements when suspended in water. Apparently in all cases lead chromate is first formed in colloidal solution and then precipitated, the rate of precipitation increasing with the concentration, *i. e.*, the amount of the electrolyte (KNO_3) in the solution. In very dilute solutions lead chromate remains in colloidal solution for several days. The rapidity of precipitation is much hastened by heating.

"Series of precipitations from solutions of varied concentrations were made at about 20° and at 90° C. From solutions of the same concentration the precipitates thrown down at the higher temperatures were uniformly darker in color, and almost uniformly composed of larger crystals. The exceptions to this latter rule were found only in the higher concentrations, where the crystals were so small that accurate measurements became practically impossible. The precipitates thrown down at 90° were much more uniform in character and apparently much less subject to the influence of accidental variations than were those made at 20°."

"The effect of the concentration of the precipitating solutions on the size of the precipitated crystals was followed by measuring under the microscope a series of precipitates produced [at 90°] as already described. In each case there was noted: (1) the length of the longest crystals in the preparation; (2) the length of the shortest crystals (excluding broken crsytals where possible); (3) the length which seemed to be approached most nearly by the largest number of crystals[2]. These three lengths are designated as the 'maximum,' 'minimum' and 'normal' respectively. It is believed that the normal length is at least a moderately exact index of the prevailing crystal size of the precipitate. The measurements are given in Table I.

[2] Not necessarily the *average* length.

TABLE I.

Effect of concentration of precipitating solutions on the crystal size of the precipitate.

Concentration in Gram Molecules per Liter	Length of Crystal in Thousandths of a Millimeter		
	Maximum	Minimum	Normal
1	4.8	0.8	2.1
1/2	4.8	1.6	2.4
1/4	3.2	0.5	1.8
1/8	2.4	0.4	1.2
1/16	1.6	0.4	1.2
1/32	4.0	1.2	1.9
1/64	4.0	1.2	1.9
1/128	4.0	1.6	2.4
1/256	12.8	1.6	6.4
1/512	6.4	1.6	3.2
1/1024	16.0	3.2	9.6
1/2048	24.0	6.4	12.8
1/4096	57.6	4.8	16.0
1/8192	24.0	6.4	12.8
1/16384	24.0	8.0	12.8

"These measurements, of course, apply only to the *length* of the crystals, that being the dimension susceptible to most accurate measurement. Rough measurements of width, and of area covered in the microscopic field, show general agreement with those made on length.

"In spite of considerable irregularities in the results, there is as might be expected an unmistakable tendency for the size of the crystal to rise with decrease of concentration. This increase of the crystal size with decrease of concentration is in a general way a matter of common knowledge, and goes hand in hand with the well-known tendency, which was also observed in these experiments, for dilute solutions to require a much longer time for complete precipitation than do concentrated ones.

"A series of precipitates was prepared as above except that to each solution there had been added sufficient white glue (in solution) to make a concentration of 10 grams per liter. These precipitates differed from the former ones mainly in the slowness with which they were formed. Precipitates were formed immediately only in the most concentrated solutions, and in the solutions more dilute than M/36 no solid precipitate was formed at

all. The solutions simply turned yellow and opalescent, remaining thus for several months. The retarding action of the glue is less marked the higher the temperature of precipitation. The precipitates from solutions containing glue are always composed of finer particles than those from ordinary solutions of corresponding concentration. In general the color is lighter."

The general results of Mr. Free's experiments on the precipitation of lead chromate were summed up by himself as follows:

(1) The largest crystals are obtained in hot and dilute solutions.

(2) The precipitates from the most concentrated and most dilute solutions are lighter in color than those from the intermediate solutions.

(3) Glue retards precipitation and causes the formation of smaller crystals.

The first and third of these conclusions are, of course, not new. They have been known qualitatively for years. There were no quantitative measurements however, and in any case it seemed necessary to have a series of experiments made under as nearly comparable conditions as possible. It might have been better to have added potassium nitrate to each solution in such quantities that the total concentration of potassium nitrate, after mixing, would have been constant throughout. This was a refinement which occurred to us, but which we did not have time to carry out.

The second conclusion was unexpected, that the precipitates from the most concentrated and the most dilute solutions should be lighter than those from the intermediate solutions. This shows that the color of the precipitated lead chromate does not depend entirely on the size of the crystal. It is probable that an infinitesimal change in the composition of the precipitate is also a factor, though this point calls for further study. In the work that has been done in my laboratory on the electrolytic corrosion of metals in salt solutions, it has always been found that the solution became alkaline in time, showing the carrying down of traces of acid by the precipitate.

In Mr. Miller's work experiments were made to see how the size of the precipitate varied when formed by what we call an electrolytic method. Cuprous oxide was taken as the pigment

to be studied because it was known that the color varied very much with the conditions of precipitations. When a cold sodium chloride solution is electrolyzed between copper electrodes, the resulting cuprous chloride and sodium hydroxide react to form sodium chloride and an orange precipitate of cuprous hydroxide. If the same process is carried out on hot, the precipitate is the red cuprous oxide. Of course, looked at in one way, this is merely a chemical precipitation resulting from the reaction of dilute cuprous chloride and dilute caustic soda in the presence of a large excess of sodium chloride. Since the reacting substances are generated electrolytically, it is rather quibbling not to call this the electrolytic production of cuprous oxide or cuprous hydroxide.

Experiments were made with varying current densities and varying concentrations of sodium chloride at temperatures between 60° and 100°. The most obvious result of the experiments was the surprising variation in the color of the precipitates from yellow, through brown, to deep red. For a while we were as pleased as Punch over this. To make sure that the light colored precipitates at 60° were really cuprous oxide and not hydroxide, they were heated for a while to 100°. Since there was no change of color, and since light colored precipitates were obtained at 100° when gelatine was added to the solution, it seemed reasonably certain that the precipitates were all cuprous oxide and that the variations in color were due to differences in physical state. Unfortunately the analyses made by Mr. Gillett did not bear out this view. The precipitates varied in composition all the way from $Cu_2(OH)_2$ to Cu_2O. They are probably a continuous series of solid solutions; but this point has not yet been established definitely and is, after all, immaterial for our present purposes. There are, however, some curious equilibrium relations, or rather lack of equilibrium relations, which I hope to straighten out some day.

Ignoring the composition of the apparently homogeneous precipitate, and considering only the size of the particles, it was found that they were larger the higher the temperature and the lower the current density. In other words, high temperature and dilute solutions give the coarser particles just as Mr. Free found with the purely chemical precipitation of lead chromate. The

effect due to the concentration of the sodium chloride was very slight indeed, and is probably to be attributed to the increased solubility of cuprous hydroxide in the more concentrated sodium chloride solution resulting in a slower precipitation and a coarser structure. Addition of gelatine decreased the size of the particles very much.

Since it is possible to vary the coarseness of a precipitate over quite a range, there was one experiment on which I had hoped to report this year. Through circumstances over which I had no control, the work was not finished during the summer, and all I can do now is to point out what might have been. Suppose we take a coarse and a fine lead hydroxide and a coarse and a fine lead carbonate. Then suppose we combine these in all possible ways to form white lead, PbO_2H_2, $2PbCO_3$. On paper we shall have four different white leads and a comparison of the densities, covering powers and oil absorptions of these four precipitates, with the corresponding values for white lead made by the old Dutch process should give us some valuable information as to what must be done next.

Cornell University.

Discussion opened by A. F. Ganz at the Fourteenth General Meeting of the American Electrochemical Society, October 30, 1908; President E. G. Acheson in the Chair.

THE CORROSION OF IRON.

General Discussion Opened by Professor A. F. Ganz.

PROFESSOR GANZ: Mr. President and members of the society: When your Secretary invited me some weeks ago to open a general discussion on the subject of Corrosion of Underground Structures, he stated in his letter that the electrochemists could probably help us in the solution of our electrolysis problems; coming as this does from an authority on electrochemistry, I felt that this was very encouraging, and I was glad to accept the invitation.

For some time past I have devoted some time to the study of the causes and prevention of corrosion of underground structures, particularly from the effects of electrolysis caused by stray electric currents. In this work I have met a number of problems which I believe can be solved by the electrochemist, and I feel that the best possible way for me to open this subject before you this afternoon will be to present to you a number of these problems in order to bring out the points which the electrical engineer meets when he is called upon to examine an underground metallic structure for its electrolytic condition, and, when a dangerous condition is found, to recommend remedial measures. Before putting these questions, however, I want to say a few words by way of introduction.

The corrosion of underground metal structures may be divided into two general classes; first, corrosion due to purely chemical causes or ordinary rusting; second, corrosion due to electrolysis from stray electric currents. Whether or not all chemical corrosion is also electrolytic in its nature does not alter this division from the standpoint of the engineer.

I am particularly interested in the second division of corrosion, namely, that caused by stray electric currents. When an elec-

trical engineer is called upon to make an electrolytic investigation of, say, a piping system, he may have two distinctly separate problems to solve. The first problem is to determine the electrical condition of the underground system, that is, the potential at various parts of the system with reference to the surrounding ground and with reference to other underground metal; he must also determine the current flowing on the underground structure, and locate the points where this current leaves the structure for ground, because at these points corrosion may develop. If in this investigation he does find that the system is in electrolytic danger, then his second problem is to recommend some method whereby the danger can be substantially removed; and that at a reasonable cost, for this is always an important element.

In regard to suitable methods for making such electrolytic tests, we have obtained considerable help from a well known German electrochemist, Dr. Haber, of Karlsruhe, who has devised an earth ampere meter and a non-polarizable electrode for measuring earth currents and earth potentials. I see, however, Dr. Sharp's name on the program for a paper on the subject of "Correct Methods of Measuring Stray Currents," and I know Dr. Sharp well enough to be certain that he will fully cover this part of the subject, so I will pass it over.

I cannot resist the temptation, however, to call attention again to one wholly incorrect method which has been used in this country in a certain class of electrolysis reports, in which the potential differences are measured between pipe and rail, and the earth resistances are measured between the same points, or, to be more correct, a resistance is measured between the pipe and rail which is called the earth resistance between these two. This resistance is then divided into the potential difference, and the current so calculated is stated to be the current flowing between the pipe and rails. This is repeated for, say, every block of a given street, and these currents are added up and the total current is stated in all seriousness to be the current which flows in that street between the pipe and rail. The absurdity of this method will be clear to you all, and I hope that every member of this society will expose such fallacious methods at every opportunity because this class of reports has done a great deal of harm in causing unnecessary alarm.

A great many reliable and accurate tests of stray currents have, however, been made, both in this country and in Europe; the results which have been published show that underground metallic structures which are continuous, as, for instance, most gas and water piping systems, do carry considerable stray currents where they are in the vicinity of single trolley electric railroads, and in many cases also where they are many miles from any electric railroad. These piping systems generally pick up current over an extended negative area and deliver this current to ground over comparatively small positive areas, producing thereby high current densities where the current leaves the pipe, causing corresponding rapid corrosion.

The question has often been asked whether the steel foundations of bridges and buildings which are usually imbedded in concrete are also suffering from corrosion due to these stray railway currents. I do not believe that we have enough real experience to answer this question positively. These steel underground foundations do not have, however, the same opportunity of picking up currents over an extended area and delivering it from small areas at a high current density as have continuous piping systems. Such structures ordinarily can be affected only by current which passes through the ground in the path of which these structures lie. There is one case, however, that occurs to me where such structures would certainly be in serious danger. Suppose, for instance, we have a pipe in metallic contact with such a structure, as is sometimes the case. If this pipe is carrying current, and is positive to the surrounding ground, a current picked up over an extended negative area may be delivered to this underground foundation structure and leave it at a high current density producing corresponding electrolytic corrosion. I believe that in a number of instances special precautions have been taken to insulate the steel foundations of structures from piping systems, and it seems to me that this would be a safe precaution to take in all cases.

The question of the maximum allowable potential difference between rails and pipes, and of the maximum allowable current in the pipe, which will not produce appreciable electrolytic corrosion, is one that has frequently come up, and in some cases municipal ordinances have been put into effect limiting either

these potential differences, or currents, or both. The old notion that a minimum potential difference of 1.5 volts is required between two iron underground structures before electrolytic action can take place has long been exploded. Nevertheless we see occasionally the statement in electrolysis reports that where the potential difference is less than 1.5 volts there can be no electrolysis from stray currents.

Where dangerous electrolytic conditions in an underground system are found, the second problem that the electrical engineer has to meet is to recommend proper preventative measures which will not be prohibitive in cost. It is in this that I believe that the electrical engineer can get the most help from the electrochemist.

The natural solution is, of course, to remove the cause of the trouble, that is, to get the electric railroads to take care of their own currents. Those of you who have actually tried to do this know that for many practical reasons this is not always possible. The electric railroad may not be willing or may not be able to make the necessary expenditures, and the business relations between the parties concerned may be such that they do not wish to compel the railroad company to do this; we therefore must frequently face the problem independently and see whether there is anything we can do to our piping and other underground metallic systems to effectively protect them from electrolytic corrosion caused by these stray electric railway currents.

If the underground metal system is continuous it may be connected to the negative return system of the electric railroad. This is, however, a method which invariably greatly increases the current flow on the system and produces other dangers, as, for instance, at a high resistance point, which may develop in a joint; here the current may shunt around such a high resistance point and produce corrosion. The greatest objection, however, lies in the danger that such a bonded system constitutes to all other underground metal systems. If we connect one piping system to the negative bus bar, we render it highly negative to all other underground metallic structures and induce current to flow to this piping system; such a bonded piping system becomes, in fact, a part of the trolley return circuit, and the owner becomes a party to any damage that may result in the other underground

systems. I firmly believe that only such methods should be used which substantially prevent the stray current from flowing to and from the underground structures.

I will now put to you five questions which embody the points that I want to present for general discussion.

Question No. 1.—This relates to the activity or passivity of iron in street soils. Are the conditions of street soils such that all underground iron may be considered as in the active state, so that we can correctly estimate the weight of iron corroded by a given current leaving such iron for ground, on the usual basis of 20 pounds of iron per ampere-year? Or do actual cases arise in practice where the ground conditions are such that iron in the ground is in the passive state, so that any amount of current can leave the iron without producing corrosion?

Prof. Haber, of Karlsruhe, who has investigated this question in European cities, concludes that under the ordinary soil conditions iron is always in the active state, and that the passive state does not exist in the underground iron structures. I would like to know whether any of the electrochemists present have any knowledge or experience in regard to this. I would like to know if there is anything we can do to render active iron passive. Someone has suggested to cover an underground pipe with lime for that purpose. Is there any practical way in which we can render our pipes permanently passive and protect them in that way from electrolytic corrosion?

Question No. 2.—This relates to means for identifying the causes which have produced a given corrosion. As a practical example of this I have a piece of pipe here which was sent to me from one of the cities in the northern part of New York State, which is somewhat corroded; as you see, the pipe is practically riddled with holes. This was a cast iron gas pipe. This sample of pipe was sent to me and the question asked whether the corrosion was due to electrolysis from stray currents, or whether it was ordinary rusting. I have another piece of pipe here, which is a wrought iron pipe, not quite as badly corroded, but which also shows decided pittings; the question here is, also, whether this pitting was caused by electrolysis from stray currents, or by ordinary rusting. I do not know of any way that would enable me to decide these questions. If any electro-chemist

here can describe some physical or chemical test which we can apply to a corroded pipe which will determine whether a given corrosion or pitting was caused by electrolysis from stray currents, or by ordinary rusting, he will help us greatly.

I have noticed some recent cases where a large amount of steel pipe which has been in the ground many years was examined and which in a great number of cases was found to be pitted. When this pipe was uncovered, scraped off with a wire brush, and washed off, the bottom surfaces of the pitting had a clean, bright metallic luster. I have seen other cases of pitted steel pipe where the interior of the pitting was full of rust, and when washed out these did not look clean and bright, but were decidedly rusty. I would like to know whether either of these appearances is characteristic of any particular kind of corrosion. I would also like to put this question: Suppose we find a pipe in the ground which is corroded and pitted, and also find by electrical tests that current is leaving the pipe in this vicinity, can we take that as positive evidence that the corrosion was caused by electrolysis from this current?

I have also some samples of corroded lead cable. Here are two pieces which were sent to me to say whether the corrosion was caused by electrolysis from stray currents, or by ordinary chemical corrosion. I do not know whether there is anything about the corrosion and pitting of a piece of lead caused by electrolysis from stray current which differentiates it from ordinary corrosion, and would also like to hear of some method of identification.

Question No. 3.—Can we protect an underground metal structure, such as a piping system, by coating it with an insulating material in such a way as to prevent current from entering or leaving the structure? A great many attempts have been made to protect pipes by painting them, by covering them with tar, and by wrapping them with cloth, as well as coating with pitch or tar, etc., but in every case that I have any knowledge of these methods have failed to protect the pipe where stray electric currents were leaving the pipe.

I have seen one case, where a steel pipe was covered with a thin layer of some tar compound, which appeared perfect when examined through an ordinary reading glass; when this coating

was scraped off, considerable force had to be applied, showing that the coating had adhered to the steel, and yet, when the steel was scraped clear, deep pittings were found in the steel under this coating. In this case the pipe was lying in wet ground, and stray current had also been leaving this pipe. The question is: Was the failure to protect due to imperfections in this covering which could not be seen, or did the corroding action of the electric current go on directly through the protecting covering?

I have recently suggested a plan for protecting a special pipe from electrolysis caused by stray currents by boxing the pipe so as to leave, say, one inch clear around the pipe and then pouring in between the box and the pipe some material like asphaltum, of a grade that will not set too hard, but will remain somewhat flexible when it has set. I would like to get the opinion of the electrochemists as to the probable success of such a coating, assuming it to be physically intact. I am informed that a similar method has been occasionally used, and that in some cases it appears to protect the pipe, while in other cases it is reported to have failed. The failure may, however, have been due to mechanical defects. This method is, of course, very expensive, and in most cases probably prohibitively expensive.

One important question comes to my mind in this connection. Let us assume that we have an iron pipe which has been in the ground for some years, and where corrosion has already started all over the pipe. Suppose we scrape the pipe off as well as we can with a wire brush and then imbed it in one inch of asphaltum. Can the corrosion which has started continue under this coating? Is it necessary to first thoroughly remove all of the oxide coating, or to paint the pipe in any way before imbedding it in the asphaltum?

You probably know that it has been attempted to use concrete to protect pipes. I know of one case where a pipe was boxed, leaving from four to five inches between pipe and box, and this space was filled with concrete; this pipe was in a location where currents were leaving the pipe, and where previously there had been considerable electrolytic action. It was found that the corrosion went on even more rapidly with the pipe imbedded in concrete than before, and the pipe was destroyed in a comparatively short time. When the box was broken open, the iron pipe

was found to be completely destroyed, and the rust had penetrated more than an inch into the contrete. If I am correctly informed, steel, when imbedded in concrete and placed under water or in ground, is protected from ordinary chemical corrosion. Why is it not protected from electrolytic corrosion? Is there anything that we can do to the concrete so that it will protect the pipe and prevent electrolytic corrosion?

Question No. 4.—Can we practically break up the continuity of an underground metal system, such as a piping system, by the use of insulated joints, so as to make it substantially safe from electrolytic corrosion, and what is the maximum allowable length of the insulated sections under ordinary conditions? Or what is the maximum allowable potential differences across an insulated joint in an underground metal system?

Question No. 5.—What is a safe density of current per square foot of pipe surface leaving for ground which will not produce substantial corrosion? I have set this figure tentatively somewhere between one and ten milliamperes per square foot of pipe surface, based on the following calculation: One cubic foot of iron weighs, roughly, 480 pounds; a piece of iron one square foot in section and one inch thick will weigh 40 pounds; the thickness of a sheet or iron one square foot in section, which will weigh one pound, is 0.025 or 1/40 of an inch. On the basis of one ampere year corroding 20 pounds of iron, one milliampere will corrode 0.02 of a pound of iron in one year. If this milliampere of current is uniformly distributed over the surface, it will corrode in one year a thickness of $0.02 \times 0.025 = 0.0005$ inch of iron, and under these conditions it would take 200 years to corrode through a thickness of one-tenth of an inch or iron. But we know from experience that current does not generally leave a pipe surface uniformly, but from localized portions producing pittings. Suppose, therefore, that this assumed current leaves from 10 per cent. of the pipe surface; on this basis one milliampere from one square foot of pipe surface would produce pittings which would have an average depth of 0.005 inch in one year, and it would take 20 years to produce pittings one-tenth of an inch deep.

This ends my list of questions, and I hope that the discussion to follow will bring out answers which will help us in our electrolysis problems.

DISCUSSION

MR. ALLERTON L. CUSHMAN: I think Prof. Ganz's address brings out very emphatically a great need that is before the scientific world to-day, the question of nomenclature—the question of being able to define what we mean when we speak of electrolysis.

Since I have been devoting a considerable amount of time to the studying of this subject, I have been brought into contact and into correspondence with a great many people all over the world, and I find that it is rare that men mean the same thing when they refer to electrolysis.

Now, Prof. Ganz has spoken to us to-day of the difference between electrolytic action and ordinary commercial corrosion. That, to my mind, is a distinction without a difference. I do not recognize any form of corrosion of any metal which is not in its true sense electrolytic corrosion.

Now, stray currents, such as have been described to us here to-day, most undoubtedly are responsible for some special cases of trouble noticed in great cities, in the vicinity of street railways and conduits that carry high potential electric currents, but if this sort of corrosion was confined to these localities, we could then indeed point to a case like this and say it is a true case of electrolytic corrosion, but it is not so confined. There was within the past two weeks towed in from Cape Hatteras one of the United States Government light ships. It had to be towed in after inspection in a great hurry to keep the ship from sinking. When it was gotten to Baltimore it was found to be pretty generally corroded in just this sort of way. It was possible with an ordinary pocket knife to take rivets and bolts out of the plates It was possible with an ordinary penknife to whittle away the surface of the plate.

I cannot understand that there should be stray currents floating around in the Atlantic ocean. I am willing to repeat that stray currents in the neighborhood of these high potential electric conduits have much to do with the ordinary electrolytic effect, but I do not believe they are responsible in themselves for the great amount of damage that is being done to iron and steel all over the world from corrosion. If one were to bury a pipe line like that out on the middle of our Dakota prairies it would suffer from corrosion in precisely the same way. In the course of time it

would be very deeply indented and rusted at certain points, which would finally give way. I do not say that in the middle of the Dakota prairie that that action would go on as rapidly as it does in the City of New York, but I do say that the same action would go on, and it is surprising, and every one who has had any experience with pipe lines knows that they go to pieces with surprising rapidity in localities where it is impossible to suppose that there are any stray currents wandering about.

So, then, I repeat most emphatically that we need to revise our nomenclature. When I speak of electrolytic corrosion, I do not mean the corrosion which is carried on by a stray current following a pipe and leaving it at some particular point. I mean the underlying and fundamental action in corrosion, in general, by electrolytic effect. The engineers, however, are apt to overlook that and to speak of electrolysis and corrosion as being a matter entirely of stray currents, and if a distinctive name could be brought out to distinguish these two kinds of electrolysis that go on, it would certainly help us and do away with a great many of our differences of opinion which occur sometimes because we do not understand exactly what is being referred to.

Mr. Maximillian Toch: I cannot answer all the questions which have been asked, because I do not know how to answer them all, and some of the questions I cannot answer now, because they are taken up in my paper, which is No. 10, but two or three of the points the gentleman has brought out I am more or less familiar with, but before I touch on them, I desire to corroborate what Dr. Cushman said with reference to the corrosion of these pipes. As far as I am concerned, it is settled in my mind that all corrosion is an electrolytic function. That is the fundamental basis of it. I do believe that stray electric currents aggravate that corrosion, and that corrosion in the City of New York will go on faster than it would on the Dakota plains. I question whether there would be any dispute on that point.

Prof. Ganz has asked with reference to gas pipe coated with asphaltum, and what the result would be. There was such an experiment made unintentionally by a natural gas company, who built a pipe line from Peoria to Chicago, and coated it to the best of my knowledge and information with asphalt about a quarter of an inch on the inside, and half an inch on the outside. The

man who had coated the pipe was not aware of the fact that fuel gas is a powerful solvent for all bituminous compounds, and in a very short time the entire pipe was so clogged up with the dissolving bitumen and bituminous compounds that it became useless, so that bitumen or asphalt or tar should be excluded from the interior of a gas pipe.

The second question is why concrete does not preserve steel against so-called electrolytic corrosion, and I will say that question was answered two or three years ago by my paper which I presented, and I believe that Prof. Bancroft still has my original exhibit showing the various brickettes of cement, starting from neat cement up to 1, 3 and 5, with pieces of steel bedded in the cement, and these pieces charged, by means of a direct current of known strength, under certain acid and alkaline and aqueous conditions. I think I demonstrated there that the so-called chemical corrosion or inhibition of the corrosion by means of concrete, cannot be avoided where certain laws of pressure obtain.

The third question which I want to bring up is the question of the pipes, which Prof. Ganz has mentioned, and he asks whether a commercial method cannot be devised for coating a gas pipe to prevent corrosion, and it is quite evident that Prof. Ganz has not had much to do with public corporations—it strikes me, no matter what system you would devise for such an effect, if it costs any more than about a mill per running foot the corporations will not accept it, because the sum total would be many thousands of dollars for several hundred miles of piping. If public corporations would undertake to pay a reasonable figure, I have no doubt methods could be invented for employing simple checks against corrosion, but on that matter I will speak when my paper comes up.

PROF. GANZ: I would like to reply to Dr. Cushman. Sometime ago I read Dr. Cushman's classical paper on "The Corrosion of Iron" with very great interest and I thoroughly appreciate that all corrosion may correctly be considered as electrolytic in its nature. The classification which I made in my remarks was not intended to be a distinction between chemical corrosion and electrolytic corrosion, but a distinction between corrosion produced by local causes without the influence of external electric currents, and corrosion produced by external electric currents. This is

an important engineering problem as the following instance which I have met in my practice will illustrate. A long pipe line passes the power station of an electric trolley road and for a distance of say a mile to each side of the station serious corrosion of the pipe takes place, producing leaks in the pipe at some point within the section nearly every year and requiring the replacement of the leaking pipe; when this pipe is removed it is found to be full of pits. The rest of the pipe line further away from the power station remains intact without giving trouble for 25 years. Electrical tests show that in the section adjacent to the power house current is leaving the pipe for ground while in the distant sections current passes from ground to pipe. I think Dr. Cushman will have to admit that the engineer can conclude that the destruction of the pipe in the vicinity of the power house was directly caused by stray currents from this power house.

DR. CUSHMAN: I do admit it.

PROF. GANZ: That is the problem we have to face. Mr. Toch suggested that I may not have had experience with municipal authorities and perhaps I have not had as much experience as Mr. Toch. Beside municipal owners there are however many private corporations owning considerable underground piping systems which are extremely important and valuable which must be protected from the destructive effects of stray railway currents.

MR. CARL HERING: I have been very much interested in Prof. Ganz's statement of the case, and I hope it will lead to some results, because there is certainly room for a great deal of good work to be done in the prevention of electrolysis by stray currents. One of the questions he asked is—can current leave the pipe without corrosion? I would like very much to hear that question discussed. It seems to me it could be answered negatively, that is, that current cannot leave the pipe for the soil (if the only conductors in the soil are liquids), without causing corrosion. If I am mistaken in this, I should like very much to be corrected.

There is one method by which corrosion of a pipe might perhaps be protected. I mentioned it some years ago before this Society. If a block of zinc is buried in the ground and attached to the pipe metalically, a current in that pipe would prefer to leave through the zinc than through the pipe. because it takes less voltage; the zinc sucks the current out so to speak; and as long as

there is zinc left the pipe would be protected from corrosion; the zinc can readily be replaced after it is consumed. If the current is very large, the cost of the zinc may be quite great. For expensive parts like motors, valves, special joints, the expense may be warranted.

As to coating pipes to prevent corrosion, it seems to me that while it may prevent electrolytic corrosion by stray currents where it keeps the electrolyte from coming into contact with the pipe, yet these coatings are very apt to be injured in spots, which means that the corrosion, instead of being spread over a large surface, will be condensed on small parts, and will therefore do all the more damage, because when condensed on a small area a current will eat through the pipe in shorter time.

Prof. Ganz asks what current density is a safe limit; by current density, I presume he meant the current density of the current leaving the pipe for the soil. I think that should be answered negatively; that is, no current density leaving the pipe is safe, because you never know whether it is going to leave from that area or be confined to a few small spots. If we were sure it would leave all over the surface of the pipe evenly, as might be the case if the pipe were laid through salt water, then there might be a safe limit, such as Prof. Ganz calculated, but in ordinary soil it does not leave in that way, but leaves where it meets the least resistance, which is likely to be in spots.

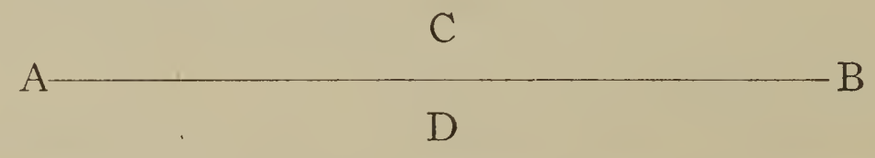
I agree with Dr. Cushman that we should distinguish between the corrosion by stray current and corrosion by what may be called local currents. What Prof. Ganz referred to I understood to mean corrosion by stray currents as distinguished from corrosion by local currents, due to impurities in the iron itself.

It has often been said that certain corrosions could not have been electrolytic, because the pipe was located too far from any electric current. It seems to me that it is very unsafe to say this. A current will travel a long way, if the resistance is low. If we have a large body of water, such as the water under the ground, it seems to me current may travel many miles, and that it is not correct, therefore, to say that because a pipe was far away from the nearest trolley line, that therefore the corrosion could not possibly have been electrolytic by stray currents.

I have noticed cases in which when a pipe was in danger at a

certain place, it was "protected" at that place, and it was thought that this was sufficient. Now, it seems to me that is very incorrect reasoning, as this so called protection is quite likely to shift the danger point to another place. I know one case in my experience, in which a pipe ran underneath the trolley tracks, crossing these tracks perpendicularly, and it was known to be a point of great danger; the pipe was then protected for a distance of twenty or thirty feet under the track by a metallic insulated casing, and it was thought that this would cure the evil; but it simply shifted the evil, as the subsequent corrosion took place at the ends of the protected section.

Concerning the measuring of the resistance of the ground, I call attention to the following experiment: If two plates are placed in the ocean, say several feet apart, and the resistance between them measured, and if one supposes that one has thereby measured the resistance of the salt water betewen the plates, one is very much mistaken, because if the plates be moved further apart it is stated by good authority that one will get just about the same value for the resistance, showing that one is not measuring the resistance between them at all, but the resistance of practically the whole ocean.



Mr. Richard H. Gaines: In the course of his remarks, Prof. Ganz stated that if corrosion occurred at D in a pipe line running from A to B, and a powerhouse C was located a few miles north or south of D., we might be sure that the trouble proceeded from the powerhouse. This does not seem to me to be necessarily true. In the case of the corrosion of the Rochester Steel conduit that I described at the last Spring meeting of the Society in Albany, the City of Rochester, with its car lines and other industrial sources of electricity is located several miles away from the northern terminus of the conduit, which is 28 miles in length. In the northern half of the conduit (nearest the city) the damage to the pipe from corrosion has been slight and confined to a low wet area less than a mile in extent. Whereas the southern half of the conduit, many miles away from any known commercial source of

electricity, has been very badly damaged, and 150 rust leaks have occurred, yet no evidence of stray currents could be detected in the southern half of the line, the difference of potential, if any existed, between the pipe and the ground being too small to be measured with the millivoltmeter. The greatest damage occurred in open meadow country, 20 miles or more from town or city. The soil, however, was nearly saturated with water which was impregnated with electrolytes. The damage to the pipe line has already cost the City of Rochester many thousands of dollars. There seemed to be no other way to account for this very serious case of corrosion than as local electrochemical action, and I called it autoelectrolysis.

Mr. I. L. Roberts: I wish in this connection to make a statement of an experiment that was made about ten years ago, something that will interest you along this line, not that we succeeded in doing what we attempted to do. Mr. Edward M. Dickerson, the famous patent lawyer, whom you all know, tried some experiments in wireless telegraphy through sea water in conjunction with myself, but we found we had so much natural electricity to deal with that we could not do anything with the currents we were trying to use from the storage battery. The experiments were conducted at Wave Crest, Mr. Dickerson's summer residence, with 500 feet of No. 8 insulated wire, and we attached to the ends of this wire, at each end, a piece of galvanized iron two feet square. We made the connections with solder, the same solder as is used in galvanizing, so that we would not have any difference in potentiality between the two electrodes or terminals. Our idea was to establish at another distant point a similar span of wire and electrodes, and see if we could not, by making a direct current pass along one set of electrodes, and by working an ordinary telegraph key, send messages to the other wire. When we stretched the first one, and put instruments in the circuit, we found a strong current flowing, which flowed only in one direction. It was a strange thing that it did not reverse at any time. This current was very feeble in the forenoon, but in the afternoon about four o'clock every day this current would rise something like 300 per cent., compared to what it was in the forenoon. We were unable to find where these currents came from, but they always flowed in one direction. We tried other

places for our experiment. We took the outfit down on Long Island Sound, to a place quite a distance out on the island, where we put in these electrodes, and we got the same current flow, and in the same direction—the same perculiarities were manifested, and the current was continuous. In the morning, about eight o'clock, it would show a certain current, I have forgotten what it was—it was considerable amperage and voltage, more than you would suspect. We then reversed end for end these devices, thinking that by so doing, that there might be some possible current which was produced galvanically by the electrodes, sea water being the electrolyte or excitement. We made this change, but it made no difference whatever, the current flowed in the same dirction, and at four o'clock in the afternoon it reached its maximum, and fell off until nine o'clock at night, when we ceased our experiments for the day. We were unable to determine what was the cause, or whether it took place everywhere along the coast or not. It is an open question as to whether it was due to electricity being transmitted through the water from some distant generator, or to some other cause.

This statement may lead to some further thought on the subject of electrolysis. As these currents were found as stated, is it not possible if two plates were buried a long distance apart and connected by insulated wire and an instrument was then put in the circuit, I believe you would find some current flowing between these two electrodes at any point on the earth.

Prof. W. D. Bancroft: It makes a difference whether we are considering the corrosion of iron pipe or the corrosion of metals in general. I can cite one case where the corrosion of a metal under ordinary conditions is different from the corrosion when you impress a current. If you take nickel and make it anode in a sodium chloride solution, the nickel will dissolve quantitatively, and you will get 100 per cent. efficiency, even with a low current. On the other hand, you can put the nickel in a sodium chloride solution and expose it to the air for any time you please, and get practically no corrosion whatever. Mr. Hering said he would be glad to be told of some cases where a current could leave a pipe without the pipe being corroded. If he will take a nickel pipe in a sodium sulphate solution, he can make it anode, and it will not corrode.

As regards the other experiment of the extraordinary resistance of the ocean, it is very interesting; but I cannot help feeling that it is fortunate that the ocean supplies the needed grain of salt.

PROF. GANZ: I ask Dr. Hering if he has actually tried the plan suggested, of attaching the two pieces of zinc to the pipe. It does not seem to me it can work, for the reason that the area of the zinc exposed to the ground is so small, and if you take a twleve-inch pipe it has a circumference of three feet, or three thousand feet of surface per thousand running foot of pipe. I do not think you could practically connect sufficient zinc to lead the current off the pipe in that way.

DR. HERING: I have not tried it with a pipe in the ground, but I have tried it in other ways, and find that as long as there is a piece of zinc attached to the iron, the current will not pass out of the iron. I realize that the exposed surface of the zinc is of importance. Of course, if you have to protect a large pipe, you must use zinc in such form as to get a large surface, and to see to it that the zinc is exposed to the same moisture as the pipe. If the zinc is placed in a dry spot and the iron in a wet spot, it would not operate as described.

MR. S. S. SADTLER: It seems to me there is a great deal of confusion among engineers when considering corrosion, where the current leaves the pipe. Lately a case came to my notice, where an electrical engineer said there could not possibly be electrolytic corrosion of some lead cable, because the indications of the ground were not favorable. I asked whether he expected the current to be leaving or entering the cable, and of course he thought of the cases where iron pipes corroded, and he thought it was the positive electrode. I said I thought the trouble was because the pipe was the negative electrode. I examined the pipe and found traces of sodium salts, and undoubtedly what happened, in my opinion, was that sodium formed on the lead, alloyed with it, and then was chemically washed out, leaving the lead in a spongy condition. The lead then soon gave way.

DR. CLAYTON H. SHARP: The topic which I am to present to you very briefly is the correct method of measuring stray currents. It is one of a good deal of importance in view of the large

values at stake, and in which there has been, as Prof. Ganz has already pointed out, considerable misconception.

In order to know whether a given system of pipes or other underground structures is in danger of corrosion due to stray currents, we must investigate the electrical conditions of such structures. Now, that is a thing which is not very easy to do. What we are chiefly concerned in finding out is this—is there at any point any considerable amount of electric current leaving the pipes? The problem is stated, but it is not so easily answered, for the reason that in general it is not possible to destroy the continuity of the system of piping in order to insert instruments, and it is not possible to insert instruments in the surrounding soil without disturbing the conditions as they actually exist in practice.

The electrical instrument which is chiefly used in investigations of this nature is the voltmeter or the millivoltmeter, and this instrument has, as Prof. Ganz pointed out ,sometimes been used with very distressing and alarming results. However, we can gain a great deal of information by the proper use of the voltmeter. We can find out the electrical condition of the pipe, and when I say the pipe, I mean anything of the sort underground, with respect, we will say, to the tracks of the trolley road.

We feel quite sure that where the trolley line and other structures are positive in potential with respect to the pipe, the pipe is quite safe. We feel that where the pipe is positive to other structures, that it may be in danger. We would be wrong to conclude that, because it is so, that it is actually in danger—for instance, it may be quite well insulated by dry sand or gravel, so that the discharge of electricity from the pipe to the soil electrolytically is prevented.

Under these circumstances, a large voltage, as shown by the voltmeter, the pipe being positive, is not an indication that the pipe is in danger, and to conclude that it is so in danger is to reach a false conclusion, and to excite unnecessary alarm.

Now, having determined by the use of the voltmeter what the possible danger areas are on a system of piping, the next step is to determine the current flowing on the pipe, and especially the the current leaving the pipe. To determine the current flowing, the most satisfactory method would be to interrupt the electrical

continuity of the pipe by inserting an insulating joint, and then by spanning that joint with wires passing through an ammeter, to measure the current directly. This, however, is often not feasible. We must consequently employ indirect methods for measuring the current. The principle one is by means of the fall of potential on the pipe. Electrical connections are made at two points on a single length of pipe to a sensitive low reading voltmeter, preferably one of high internal resistance, and the indications of this voltmeter or millivoltmeter are read over a sufficient period of time to show what the average condition of the pipe is. Then, if we know the resistance per unit length of pipe, it is possible to interpret these millivolt readings in terms of amperes of current flowing on the pipe. The difficulty with this method is the determination of the resistance per unit length of pipe. Now, pipes are made in standard sizes and of material which varies a good deal, but which has fairly fixed average values of resistance, and since it very often happens that we are concerned only with rather rough quantitative values, we may assume from the known dimensions of the pipe, and the known average resistance of the material, the resistance of the length of pipe with which we are dealing, and in this way make our interpretations of current.

It is possible sometimes to get at a rather better value by resorting to a procedure which consists in attaching to the pipe heavy leads, which are brought and connected to an ammeter. The potential difference between two portions of the pipe close to the place where these heavy leads are brought out is measured when the heavy leads are not connected, and again when they are connected through the ammeter, and at the same time the value of the current, as shown by the ammeter is measured. In the second case, when the ammeter is in circuit, the potential difference as shown by the millivoltmeter will be less than in the first case, and the reduction of voltage due to drawing a certain measured current from the pipe gives an indication of the resistance of that length of pipe.

For instance, if we find that by connecting the heavy leads we reduce the potential difference between the two points of the pipe to one-half of its former value, we know that the original current flowing through the length of pipe was twice what the ammeter indicated.

I have said that these millivoltmeter measurements should be made preferably with an instrument of high resistance. Except for the rapid fluctuations of voltage which occur, and fluctuations of current which ordinarily occur, a potentiometer would be a more suitable instrument to use. However, the problem not only involves the determination of the current flowing on the pipe, but more particularly the current which is escaping from the pipe. How shall we determine that. The only way I know of doing that, without seriously disturbing the normal conditions of the pipe, is by taking simultaneous measurements of the current flowing on lengths of pipe separated from each other by suitable distances. These may be taken by suitable instruments, the observers reading at the same instants, or by sensitive and accurate recording instruments, which are also obtainable suitable for this purpose. The interpretation of these results will give at least a rough measure of the current which is leaving the pipe at points between.

We can also get some indications of the probable escape of current from the pipe by exploring the electrical condition of the soil in its vicinity. If we start from the pipe, and find a continuous fall of potential as we go from point to point away from the pipe, the indications are that current is flowing along such lines, and that these lines are stream lines of electricity. In making measurements of this kind it is advisable of course to use electrodes not subject to error due to polarization, that is electrodes of zinc in zinc-sulphate solution, or similar electrodes, and make the measurements by a method which does not draw any current, so as to eliminate resistance drop at the point where the electrode is inserted in the soil, that is, some form of potentiometer is indicated as the proper instrument here.

Prof. Harber's earth ammeter, as it is sometimes called, is also used for this purpose. This consists of two sheets of metal close together enclosed in an insulating ring, and each one in contact with a salt of the same material, so they will not polarize. This arrangement is inserted in the soil close to the pipe, and is covered with the soil as nearly as possible in the same condition as it was previously to the inserting of the ammeter. The plates are connected to leads which come up through the ground, connected to an ammeter, and the ammeter indicates the current which would

flow through the displaced cross-section of the soil. The insertion of this earth ammeter changes the condition of the soil surrounding the pipe, and the indications are rendered somewhat uncertain on account of such alteration of conditions in the soil which invariably and inevitably follows with its use.

MR. CARL HERING: Dr. Sharp referred to a method I am interested in, namely, to shunt a piece of the pipe with a low resistance, and then measure the current in that low resistance, at the same time measuring the difference in drop. The method is an interesting one on paper. I was interested in it myself some time ago, until I figured what the necessary conditions must be, when I found that, unless you get very large copper conductors and excellent connections with the pipe, it would be almost impossible to carry it out, except perhaps for very small pipes. I had great hopes that this method would help us very much, but I am afraid that it is impracticable.

MR. C. F. BURGESS: I should like to hear Dr. Sharp answer Mr. Hering's statement.

DR. C. H. SHARP: I think I shall have to agree with Dr. Hering—it is frequently impracticable to carry it out. Sometimes it can be done, but in all this work we have to resort to methods that fit the case in hand, and we cannot always take the one we would like to take. Very frequently the one we think would be the best will not work at all, and the whole matter is in a rather unsatisfactory shape, because it is so frequently impossible to make exact measurements. However, measurements, even inexact ones, are of the greatest value in locating trouble from electrolysis. We have to be very content often with something which is not as good as we would like to have, but which perhaps, after all, is as good as the necessities of the case require.

DR. HERING: Have you tried the method of shunting?

DR. SHARP: I have tried it in some cases, and found it to work, and in other cases I have found it impossible to work.

DR. HERING: In what case would it work?

DR. SHARP: Where the resistance of the pipe is moderately high.

MR. S. S. SADTLER *(Communicated):* A similar occurence as far as the concentration of sodium ions, due to electrolysis in the earth, came to my notice several years ago. The case was

that of a small switchboard that had been placed on a concrete foundation wall and a white deposit seemed to have formed on it, chiefly about the negative bus bars and caused a short circuit. I diagnosed the case as that of the deposition of sodium and the formation of caustic soda and afterwards sodium carbonate by chemical action. This deposit had sufficient conductivity to cause a short circuit.

A very small percentage of sodium chloride which is always present in the earth can cause the concentration of a very large deposit of sodium ions and when the water in contact with the improvised cathode is fairly well saturated with sodium hydrate, metallic sodium can collect. I have been credibly informed that several pounds of the metal have been found at such a cathode surface underground.

It seems desirable to protect lead cables from being negative poles more than positive.

Mr. John C. Parker *(Communicated):* In regard to Prof. Ganz's queries:

1. Building foundations would apparently come under this head. These are virtually isolated and further isolated from one another than from any main paths of current likely to exist in streets, so that the at times anticipated dangers from electrolytic corrosion should not be looked for in such cases.

3. Aside from technical possibilities it would seem to be quite impracticable to do anything in the way of coating. Could the coating be made absolutely perfect, mechanically, it *might* be efficacious but defects must, in the nature of things, occur and such insulation from earth would appear to aggravate difficulties rather than to ameliorate them, except for the more close localization which renders repairs more easy. This may cover in part the suggestion in No. 1 with regard to the use of lime and such materials.

4. The breaking up of a system by means of insulation joints is believed to be dangerous rather than otherwise, as it is always the point at which current leaves the structure that is the victim of the most serious deterioration. Were it commercially practicable to break structures up into very short lengths such a system of insulation might do some good as the total resistance of structures could be made sufficiently high so that no great magni-

tude of current could be carried, but this would involve interminable difficulties with extremely special forms of joints which might readily interfere with the practical operation of the structure. Gas, water and steam distribution companies have tremendous difficulty as it is with joints of very simple construction so that not much is to be hoped for in the way of frequent interruption of metallic continuity.

MR. A. A. KNUDSON: I would add one other question to the question box which Mr. Ganz presented, and perhaps that can be attended to later in the transactions. The question is this—I have in mind at the present time two wrought iron water pipes which are undergoing corrosion probably from chemical processes. These pipes run in a creek partly submerged. The pipes are negative to the soil, at that point. There is a current of electricity flowing through it, sometimes reaching seventy-five and eighty amperes. The question I wish to ask is—will this current of electricity flowing through these pipes tend to assist the electrolytic action of corrosion on the surfaces from the chemical effect?

Paper read at the Fourteenth General Meeting of the American Electrochemical Society, New York City, October 30, 1908; President E. G. Acheson in the Chair.

THE FUNCTION OF OXYGEN IN THE CORROSION OF METALS.

By Wm. H. Walker.

The electrolytic theory of the corrosion of metals, from which point of view the role played by oxygen may best be discussed, has received the endorsement of so many able investigators working entirely independently, that further argument in its support would seem to be unnecessary. In volume two of the Journal of the Iron and Steel Institute of Great Britain for 1908, however, there appears as a report of a Carnegie Research Scholarship an article on the Corrosion of Iron, by J. Newton Friend, in which the author proves to his apparent satisfaction that "the electrolytic theory of the corrosion of iron is untenable." Were it not for the authority which publications in the Journal of the Iron and Steel Institute carry, this communication would scarcely deserve even a passing notice. As shown later, the reasoning employed is superficial, and the experiments described are untrustworthy.

When a metal is placed in water or in an atmosphere sufficiently moist so that a film of water condenses on its surface, the action which may take place is essentially one of solution. Every metal has a tendency to pass into water solution in the ionic form, assuming a positive charge of electricity and leaving the mental negatively charged. To maintain electrostatic equilibrium, an equivalent amount of positive electricity must leave the solution by the separation of hydrogen ions from the dissociated water in the form of hydrogen gas, charging that portion of the metal on which the hydrogen separates positively, and leaving the solution negatively charged. An electrolytic current is thus produced which is carried from one point on the iron to the solution by the escaping iron ions, and from the solution

again to the iron by the separating hydrogen ions, and equilibrium again restored. The speed of this reaction depends, *first,* upon the escaping tendency of the metal itself, measured by its solution pressure; *second,* upon the concentration of the hydrogen ions, increasing as this concentration is increased; *third,* upon the ease with which deposited hydrogen ions can assume a gaseous state and escape or be removed from the metallic surface.

The solution pressure of the different metals, that is, their tendency to assume an ionic form and pass into solution, varies from that shown by sodium or potassium, where this escaping tendency or solution pressure is so great that instant decomposition occurs, to that of platinum or gold, where we find the solution pressure so small that there exists no tendency to assume the ionic form. Between these two extremes we find all of the other metals, including thereunder the element hydrogen. For the purpose of the discussion of the electrolytic theory of corrosion and the part which oxygen plays therein, we can separate the metals into two divisions, viz., those which have a solution pressure less than hydrogen, as, for example, copper, lead and mercury, and those which have a greater solution pressure than hydrogen, such as magnesia, manganese, zinc and iron. So far as this one factor is concerned, therefore, metals should be decomposed by water in proportion as their solution pressure is greater or less than that of hydrogen; but other factors enter into the reaction which introduce some important exceptions to this generalization.

The second factor influencing the rate at which a given metal will pass into solution in water and thus itself be decomposed, is the concentration of the hydrogen ions. In consequence of the positive charge which the solution assumes by virtue of the positive charge of the metallic ion which has entered it, there is set up with respect to the metal surface an electrolytic double layer which arrests further progress of the solution. If there be present in the solution, however, hydrogen ions, and the metal under discussion has a solution pressure greater than that of hydrogen, ionic hydrogen will pass over into the molecular condition, giving up its positive charge to the metal, and leaving the solution negatively charged. An electric current will pass, thus breaking down the electrolytic double layer and produce

an electrostatic equilibrium which enables then this cycle to be repeated. The result is that the metal continues to pass into solution and molecular hydrogen continues to be deposited on the metal other than the point at which it is dissolving. The ease with which the hydrogen ions can neutralize or destroy this electrolytic double layer depends on their concentration, and varies from the case of pure water, where the action is excessively slow, to that of a mineral acid, in which the speed of the reaction is so rapid that hydrogen is set free from the surface of the metal as a stream of gas. Hence in the case of a metal in contact with water any reagent which increases the hydrogen concentration will accelerate the corrosion, while, on the other hand, the reagent which will decrease this hydrogen concentration will diminish the rate of corrosion. Thus does the electrolytic theory of corrosion account for and explain the increased solution, or, as we term it, corrosion of the metal by such materials as are themselves acids, or which by hydrolysis, as, for example, magnesium chloride at high temperature, furnish an acid on the one hand, and the protective or inhibitive action of such materials as milk of lime, caustic soda or cement, which, by introducing a large number of hydroxyl ions into the solution, cause the hydrogen ions already there to disappear, on the other hand.

Mr. Friend, in the paper noted at the beginning of this article, rests his conclusions that "the electrolytic theory of the corrosion of iron is untenable" upon the alleged fact that iron will not corrode in the presence of water and oxygen so long as carbon dioxide be absent, but that in the presence of water and oxygen and the most minute trace of carbon dioxide the solution of the iron with subsequent further oxidation and the formation of rust takes place. In the crucial experiments of Mr. Friend, the iron under observation was first washed with caustic soda, and the assumption made that several rinsings with pure water is all that is necessary to remove the caustic soda. We have shown by conductivity experiments that caustic soda is held by the iron most tenaciously. It is worthy of note that the one experiment in which rusting always took place in spite of every precaution to exclude carbonic acid, was the one in which the iron had not been soaked in the caustic soda previous to its

exposure. Assuming, but not admitting, that the experiments of Mr. Friend satisfactorily show that his conclusions are true, this fact would not in the slightest way contradict or even open to question the electrolytic theory of corrosion, but would, on the other hand, substantiate it. It is conceivavble, though I do not think possible, that the hydrogen concentration which obtains in pure water may not be sufficient to produce an osmotic pressure of hydrogen which in connection with the solution pressure of the iron will set up an electromotive force insufficient to cause an appreciable electric current to be generated and flow between two separate points on the surface of the iron; and that it requires just the increase in the concentration of the hydrogen ions which is occasioned by the introduction of this small amount of carbon dioxide in the form of carbonic acid to produce an electromotive force sufficient to cause such an appreciable current to flow and to thus produce the observed solution of the iron and, in the presence of the oxygen, rusting. But it will be clear that by the electrolytic theory of corrosion we are able to explain these results more consistently than can be done by any other theory of corrosion yet suggested. In fact, there is no explanation for the influence of carbon dioxide in corrosion other than the fact that it is an acid; this in turn assumes an accelerated action due to an increase in the concentration of the hydrogen ions, and we are forced to the electrolyte theory for a satisfactory explanation of the action in the end.

The third factor is the ease, due to lack of resistance, with which the electric current, generated by the solution of the metal at one point and the separation of the molecular hydrogen at another, can pass from one of these points to the other. They may be infinitely close together, or separated by quite a distance. Therefore, this theory demands that solution or corrosion should take place more rapidly in water in which there is dissolved a trace of an electrolyte, then in absolutely pure water.

As a corollary of the last feature mentioned, viz., the resistance of the external circuit, follows the influence exerted upon the rate of reaction by the ease with which the molecular hydrogen which separates or plates out upon the metal, may be removed. The resistance of a film of gas is relatively very large, and, if this hydrogen gas be not removed from the surface of the metal, the

resistance thus introduced in the circuit may be so large that no appreciable current can pass, and therefore the reaction would apparently cease. It has been long known that hydrogen is much more easily liberated from the surface of certain metals, such as copper or platinum, than it is from certain other metals, as zinc or iron. It is also a recognized fact that hydrogen will unite with oxygen to form water much more easily from the surface of certain metals than from others. Hence, the electrolytic theory demands that the solution or corrosion of the metal should vary, other things being equal, with the ease with which hydrogen may be liberated from its surface, and also with the ease or to the extent to which the reaction $H_2 + O = H_2O$ is catalyzed.

One fact regarding the corrosion of iron appears to be undisputed, viz., that oxygen is necessary for a continued action. This corrosive action can cease from two causes, viz., the osmotic pressure of the dissolved iron may increase until it neutralizes or compensates the solution pressure of the metallic iron; or the action may be stopped by the separation of a film of molecular or gaseous hydrogen upon the metal, which, owing to its resistance, prevents the flow of an appreciable current. From the fact that iron possesses, even in relatively concentrated solutions of iron salts, a very appreciable potential, it would seem highly improbable that the solution of the iron in water should be stopped by the osmotic pressure of that already dissolved, and therefore, although the oxidation and subsequent precipitation of the already dissolved iron is the most *striking* function of the oxygen, it is probably by far the least important, and its real accelerating action lies in the destruction of the hydrogen film already separated out on the surface of the metallic iron.

That this is true may be seen by the consideration of a number of now well known phenomena. When a piece of iron is placed in ordinary water exposed to the air it will dissolve or corrode or rust. If now there be placed in the water with this piece of iron a piece of platinum the solvent or corroding action of the water will not be changed. The oxygen is present in the solution as before, and the iron ions as they separate from the metallic iron are being oxidized and precipitated as rust. If now the platinum and iron be electrically connected, a marked increase

in the rate of the solution or corrosion of the iron is noticed. No chemical condition has been changed; the difference lies in the fact that there is now an electrical contact between the iron surface and the platinum, and the platinum furnishes a surface on which the hydrogen can deposit, and on which, by virtue of its catalyzing action, the hydrogen will be rapidly oxidized by the dissolved oxygen and thus removed from the sphere of action. Or, if in two limbs of a U tube separated at the bottom by a jelly plug there be placed two pieces of the same iron, electrically connected, and in a very dilute solution of any electrolyte, and both of these limbs be protected from the air, no action will be noticed. That is, there will be, on both pieces of iron, points at which iron has gone or tends to go into solution, and points at which a film of hydrogen has deposited, and owing to the resistance of this film of hydrogen no local currents can pass over the surface of the pieces of iron and no current as a whole passes through the jelly plug at the bottom, or through the system as a whole. If now there be placed in one of the limbs a substance which will unite with hydrogen, such as potassium bichromate or alkaline hydroxylamine, still no action will be observed in that limb, but the iron in the other limb will pass into solution and a current of electricity will pass through the system.

A third example is found in the action of zinc in water containing an electrolyte. If a strip of zinc and a strip of iron be placed in water containing a very little salt, the iron will corrode rapidly, while the zinc will not be noticeably attacked. The usual explanation for this phenomenon is that the zinc protects itself with an adherent film of zinc oxide or hydroxide, while the iron produces a non-adherent voluminous hydroxide which does not protect. If instead of being separated the two strips of metal be placed so as to touch each other, the iron no longer corrodes, but the zinc very rapidly passes into solution. The presence of the iron cannot affect the nature of the coating of zinc hydroxide which in the previous case adhered thereto and protected, according to the old explanation, the sheet of zinc, nor can the presence of the zinc affect the oxidation and precipitation of the iron ions should they pass into solution as they are wont to do. The explanation consistent with the electrolytic theory is that the zinc in the first arrangement does not dissolve

and corrode, not because there was formed an adherent and protecting layer of zinc hydroxide, but because the surface of the zinc does not catalyze the reaction $H_2 + O = H_2O$ with sufficient rapidity to continually remove the hydrogen from the surface of the zinc. The film of hydrogen, therefore, forms on the zinc and protects it, and it may be said to be "polarized!" Although the catalyzing action of the iron surface is not so rapid as is the platinum surface, it has sufficient rapidity to depolarize the iron strip and to render continuous the solution, and therefore the corrosion of the iron. While the solution pressure, that is, the tendency to pass into solution, is much stronger in the case of the zinc than it is in the iron, the iron corrodes and the zinc does not, because the electrolytic double layer set up on the surface of the zinc cannot be destroyed by a flow of electric current. When the two are united, the greater solution pressure of the zinc forces a larger number of zinc ions into the solution than does the iron force iron ions into the solution, and the next result of the flow of current is that the zinc soon becomes anodic over its entire surface, while the iron becomes the cathode, and that no iron is then dissolved, but as a cathode furnishes a surface on which the hydrogen set free by the solution of the zinc may be deposited, catalyzed and removed.

It has been shown by Richard and Lownes[1] that when an electric current at low current density is passed through water in the presence of air, that hydrogen is not liberated at the cathode as a gas, but that it unites with the dissolved oxygen at the electrode, forming first hydrogen peroxide, and then water. Since identically these conditions obtain in the corrosion of metals, this same phenomena doubtless takes place; in fact Dunston[2] was able to identify the presence of hydrogen peroxide in the products of the corrosion of zinc, although no direct proof of its existence on the surface of corroding iron could be obtained.

I have already shown that "mill scale" or magnetic oxide of iron is strongly electronegative to iron. Since mill scale is insoluble in water and cannot of itself enter into the reaction, its only function can be analogous to that of platinum or other insoluble conductor of this kind, viz., to furnish a surface on which the hydrogen liberated by the dissolving iron can separate and

[1] Z. Phys. Chem., 20, 145.
[2] Chem. Soc. (London), 84, 1584.

be catalytically oxidized to water again. This is also true of the black oxide protective coatings sometimes used upon iron and steel, as, for example, that of the Bower-Barf process. Just as is the case in mill scale, these coatings are very serviceable so long as the whole coating is intact. But so soon as a portion of the metallic iron is exposed, this portion corrodes all the more rapidly on account of the presence of the surface of scale on which the oxidation of the hydrogen and consequent depolarization can go on. The inevitable result is that a "pit" forms at the exposed point and grows deeper and more marked in proportion as the scale is dense and closely adherent to the iron surface. Hence, if it were possible to remove the mill scale entirely from steam boiler tubes, for example, pitting would be largely eliminated, and the life of the tube prolonged. The influence of the presence of mill scale on iron may be seen from the following experiment. Two pieces of iron were cut from a bar which was coated with the original rolling mill scale. On one of the pieces parallel strips of fresh iron were exposed, leaving alternate strips of the scale. The other piece was planed free from scale. Both were exposed to corrosion in water for one week, and the loss in grams was, for the average of a large number of tests:

Plain Steel.	Steel and Scale.
0.560 gram.	0.860 gram.

The well known fact that zinc in electrical contact with iron will protect the latter from corrosion has already been spoken of. Although this statement is universally made in text books, the fact that, in consequence of the protecting action to the iron the zinc itself will rapidly be dissolved, is not mentioned. Both actions will be the more complete, the more energetic is the solution of the zinc and the correspondingly greater flow of current from the zinc to the iron. But the flow of current will depend, among other things, upon the resistance offered to its passage through the water. Hence it should be possible to get water so pure, or to place the iron so far from the zinc that the electromotive force of the system could not overcome the resistance and no appreciable current would flow, and the iron should corrode as though no zinc were attached to it. It was found that Boston water as drawn from a tap in the laboratory had a sufficiently

low conductivity, so that zinc attached to a piece of sheet steel would protect it when placed in this water for only a distance of three-fourths of an inch. When a little common salt was added to the water, the protection was extended for a foot and one-half or more. Hence, in order that zinc may protect iron from corrosion, it is necessary to have present in the water surrounding the two, enough electrolyte to insure a low resistance to the flow of the current generated by the solution of the zinc.

The fact that the presence of a metallic iron surface accelerates the corrosion of zinc has an important bearing upon the protection of such structure as barbed wire fencing, or whenever both an iron and zinc surface are exposed. In making barbed wire fencing the wire is first galvanized and then wound into fencing, the cross-wires forming the barbs being cut at an angle in order to make a pointed barb. This freshly cut surface of iron is thus exposed, and so furnishes just the surface required for depolarizing the zinc surface and thus greatly increasing the rapidity with which the zinc coating will corrode. At the zinc coating thus recedes from the iron surface, the latter rusts on account of the low conductivity of the water film on the two, and hence the explanation of why deterioration of a barbed wire fence always starts at the barbs and travels from there along the strands.

It would seem worth while in this connection to pass the finished barbed wire through a wet galvanizing bath in which the wire should be cathode and be surrounded by a zinc anode, so that the current would be attracted to the barbs and hence the zinc deposited almost exclusively on just the points where it is needed. Little if any would be plated on the wire strands.

In examining barbed wire fencing which has been exposed for two or three years, it is not infrequently found that one of the two main unit wires making up a strand will have corroded much faster than the other which is close alongside of it and in direct contact with it. Three factors may enter into this difference. First, the steel may itself be inferior in one wire; second, the zinc coating may be thinner, *i. e.*, "wiped" closer on one than on the other; and third, the zinc coating may be contaminated with a zinc iron alloy. While the first two are matters understood by the wire makers, the third is not so easily detected. The alloy known as "hard dross" by the galvanizers, and which,

owing to its high melting point, settles to the bottom of the galvanizing pot, contains from 4 to 6 per cent. of iron. This is in reality a mixture of the alloy Zu_7Fe and zinc, and, while the properties of zinc still predominate, the effect of the presence of the alloy may be easily detected. Contrary to what might be expected, the iron zinc alloy Zu_7Fe is *electronegative* to iron; that is, it will act like platinum or mill scale and accelerate the depolarization of the cathodic (hydrogen) surface, and thus aid corrosion. This alloy and its behavior towards oxidation will form the subject of a future paper, but its influence on the ease with which hydrogen may be liberated from a zinc surface may be seen from the following experiment.

Alloys of iron and zinc were prepared and pieces of a uniform size placed in 2 per cent. H_2SO_4 for ½ an hour. The loss of weight was determined and is given in grams loss per minute per sq. in. exposed area.

% Fe in Zinc	Total loss in weight	Loss per minute
0.60	0.240 gram.	.0040
1.20	0.384 "	.0062
1.90	0.504 "	.0084
2.66	0.658 "	.0109
3.35	0.694 "	.0115
3.80	0.742 "	.0123
4.40	0.852 "	.0142
4.90	0.888 "	.0148
5.50	0.925 "	.0154
7.00	0.971 "	.0161
...	1.074 "	.0179

The concentration of the hydrogen ion in such a case as this is so widely different from that which exists in water as we ordinarily find it, and oxygen plays no negligible a part in this acid solution compared with ordinary corrosion, that I think a comparison here of doubtful value; but the table shows the direction which the effect produced by the presence of such an alloy would take.

Since corrosion depends upon the depolarizing effect of oxygen, any reagent which would exclude oxygen from water should retard corrosion. It has been shown that the solubility of oxygen in a solution of common salt decreases as the content of salt increases. Hence the corrosion of iron should be inversely proportional to the concentration of the salt solution. In the

following table are given the loss of iron in grams per sq. in. exposed area of some steel plates exposed alone, and the loss of both steel and zinc plates where the two were in contact.

IRON IN WATER CONTAINING SALT.

Water contains NaCl	1 wk. exposure	2 ½ wks. exposure
25 Gm. per L.	.0786	.175
50 " " "	.0698	.150
100 " " "	.0614	.132
150 " " "	.0454	.101
	.0341	.065
250 " " "	.0180	.057
300 " " "	.0068	.045

IRON AND ZINC IN CONTACT.

H_2O	Fe	Zu
10	.032	.097
25	.0004	.170
50	.0007	.163
100	.0005	.154
150	.....	.130
250	.....	.087
300	.....	.062

A common type of corrosion of iron which is generally considered as a subject by itself is the corrosion of tubes and shells of steam boilers. The function of the oxygen (with which the normal feed water of a boiler is saturated) is here the same as in other types of corrosion, and, as boiler tubes cannot be protected by paints or other coatings, the detrimental effect of the oxygen must be counteracted in some other way.

If the feed water used be perfectly free from dissolved salts, there is lacking in the boiler that protective coating of scale which, while it reduces the heating efficiency of the boiler, also protects the iron of the boiler from the access of oxygen. In such a case it is well to feed into the boiler, when first put into commission, enough water containing scale forming salts to thoroughly coat the shell and tubes with a layer of hard boiler scale. This practice is very common in marine boilers which are to be fed with distilled water. Although, as we have seen, the presence of alkalies inhibit the corrosion of iron by reducing the hydrogen concentration, and although, as Dr. A. S. Cushman has shown, the presence of bichromates in solution greatly inhibits this ac-

tion, the best practice is to remove oxygen from the feed water before it is introduced into the boiler, just as it is the best practice to remove the scale forming salts of the ordinary feed water before this is used.

As the solubility of oxygen in water rapidly decreases with rise in temperature, by far the larger portion of the oxygen dissolved in natural waters, or in distilled water, is removed by heating the water to near its boiling point. If, in addition to the application of heat, a vacuum be maintained over the water, the dissolved oxygen is pracically all removed, and such feed water will never corrode the boiler. Such an arrangement has been in use in a few of our largest steam generating plants with the greatest success. Even if no diminished pressure can be maintained over the hot feed water, the mere heating to the boiling point will drive out most of the dissolved oxygen. If, therefore, an open feed-water heater be employed, the feed water is deprived of its oxygen without additional expense for the heat, because the feed water in any case must be brought to its boiling point the moment it gets within the boiler. This method of treating boiler feed water has also been found very efficient in the power stations of some of our large electric railway companies.

A third method of depriving the feed water of its oxygen is to carry the same through a tank filled with small pieces of scrap iron. The accumulated iron hydroxide must of course be cleaned out of the tank periodically and the scrap iron renewed. But from the point of economy it is of course much cheaper to pit and corrode scrap iron in a tank than it is the tubes inside the boiler, so that this method, especially if combined with an open feed-water pre-heater, is in the end a very economical and highly efficient one.

If, for any reason, it is not possible to remove the oxygen from the water before it is fed into the boiler, numberless boiler compounds are proposed to counteract the effect of the oxygen within the boiler. Such a method is of course irrational, and is nothing more than voluntarily taking poison with the expectation and intention of taking at the same time an antidote therefor. The compounds and mixtures which have been proposed for introduction into a boiler include practically every thinkable material extant. The basis of most of them, however, is sodium

carbonate in the form of soda-ash, and are effective only insofar as they reduce the hydrogen concentration in the water. A considerable portion of the soda added is frequently consumed in precipitating the scale forming salts of the water, and is as a rule of low efficiency as an anti-corroding ingredient. Inasmuch as an alkaline solution of the ordinary tannins or tannin extracts breaks up more or less when subjected to the heat and pressure of a steam boiler into gallic and pyrogallic acids, which are energetic absorbents of oxygen, a solution of this material is efficient as a boiler compound for preventing corrosion.

A very old method is, of course, the introduction of pieces of metallic zinc into the boiler, which operate in accordance with the principles already discussed. It is necessary, however, to maintain good electrical contact with the iron and to have in the water sufficient electrolyte of some sort to carry the current from the zinc to the iron. For the protection of condenser jackets using sea water this method has been found to be perfectly satisfactory and entirely trustworthy.

Research Laboratory of Applied Chemistry,
Massachusetts Institute of Technology,
October, 1908.

ELECTROLYTIC CORROSION OF THE BOTTOM OF OIL TANKS AND OF OTHER STRUCTURES

By Adolphus A. Knudson, Electrical Engineer.

The subject of the corrosion of iron in its broad sense being of wide and growing interest at the present time, any data furnished of special or important features of corrosion discovered in the practical operation of various industries will doubtless be of some value, not only to those engaged in laboratory studies of the question searching for cause and remedy, but those directly concerned who are seeking to avoid the depreciation of costly structures.

In accepting the invitation to prepare a paper for this meeting, the writer has felt a sense of duty as a member of the society to bring before it at this time for discussion, a case of most pronounced corrosion which has recently come under his notice, and which, it seems plain, comes squarely under the head of electrochemistry.

The case to which we refer is the destructive effects found upon the bottom of oil tanks. Three such tanks have been examined and are the principal subject of this paper. Two of these are located in the gas works of an eastern city and one in the Standard Oil Works of another city.

First, a word in regard to the construction of oil tanks. This being fairly well known, a brief description only is necessary. The many different sizes are made of riveted sheet steel plates, usually 11 feet x 5 feet and from ¼-inch to ⅜-inch in thickness for the sides and bottoms, and somewhat thinner for the roofs. The roofs are supported in some cases by upright wooden joists from floor to roof, the joists suitably braced. In other cases a steel frame is placed under the roof supported from the sides, similar in appearance to an umbrella frame.

The conditions found in the three tanks examined will now be considered. First, the two tanks at the gas works. These will be considered together, as all the conditions were practically the same in each case, even to the extent of damage found.

Fig. 1 shows the top view in section of both tanks. These were found numbered 7 and 8 (the same numbers used in the drawing), and will be herein so referred to. These tanks are 35 feet in diameter and 16 feet high, slightly higher at the peak of the roofs. The roofs are supported in this case by perpendicular wooden joists from the floor, as shown by the circle of square dots in the drawing.. An intake pipe is located

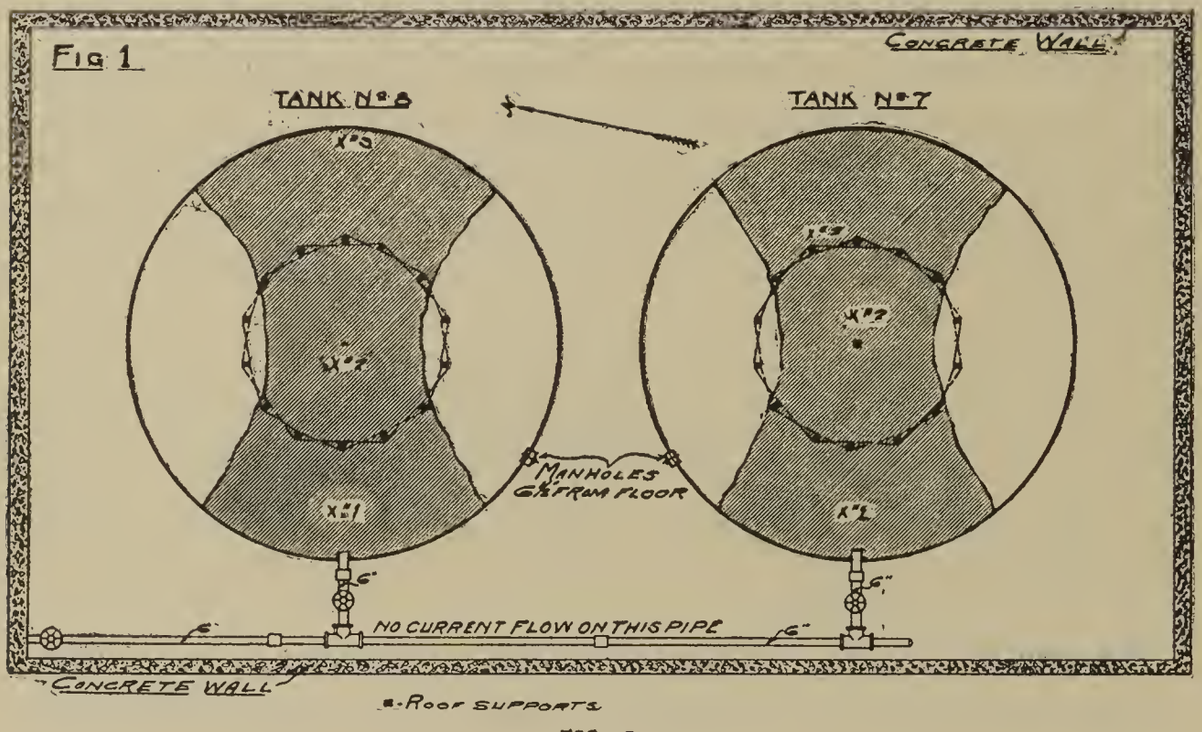


FIG. 1.

about 6 inches from the bottom of each tank, which is also used as an outlet. These intakes connect to the outside pipe line shown in the drawing.

These tanks which are located on the end of a dock are enclosed within a concrete wall, with concrete foundations; this presumably to prevent oil flowing into the harbor in case of leaks or fire. Both of these tanks were discovered to be leaking. The oil was run off and a sediment of a consistency of soft soap removed. The numerous pittings and holes in the bottoms suggested to the men at first, electrolysis due to railway currents, as it was known such currents were flowing through the

works. When the writer was called to examine, the first discovery made was the pittings originated at the *interior* of both tanks, the larger part of such pittings on the inside floor, and tapering downward, many of them terminating in holes through the iron. These holes ranged in size from ¼-inch in diameter to more than 1-inch in several cases.

Plaster casts were taken in a few places on the floors of both tanks, so these effects could be later considered. These casts are herewith submitted for your inspection. Plaster casts, it is well known, are the reverse of holes and pittings. Photographs of these casts have been made and appear in the paper. The shaded portions of Fig. 1 show the location where these pittings

FIG. 2.

and holes were found most numerous. It will be noted they were immediately in line of the intake pipes and spreading out at the opposite sides. The numbers on the casts correspond with the numbers in the drawing, indicating where they were taken. Each of the casts will be briefly explained.

The first three are from Tank No. 7, Fig. 2.

Cast No. 1.—A pitting with a projection near the center, representing a hole through the metal ¼-inch in diameter. This is the only projection on the casts representing holes which remained intact; the others broke off when the casts were removed. At the left of this pitting are two white spots which represent holes, where the plaster broke off.

Cast No. 2.—A pitting at the far side nearly through the iron. This was taken about the center of the tank.

Cast No. 3.—A cluster of pittings representing a fair average of those discovered at the opposite side of the tank from the intake.

Plaster Casts from Tank No. 8. Cast No. 1 A group near the intake, which represents a fair average at this point, excepting some holes not far away.

Cast No. 2. Shows a ridge representing a furrow or channel in the iron bottom 4 inches long, 2½ inches wide and 6-32 inch at the deepest point, or within 2-32 inch of being through the iron. In the immediate vicinity of this cast were found several holes.

Cast No. 3. The top broken off, representing a pitting with a hole at the center. This hole was 1 inch long and ½ inch wide, measuring from the longest and widest parts.

These two tanks have been in service seven years. As they were much needed for storing oil, temporary repairs have been made by filling the pittings and holes with litharge and red lead, but it is probable that new bottoms will soon be placed in them.

Samples of the water obtained from the bottom of two of

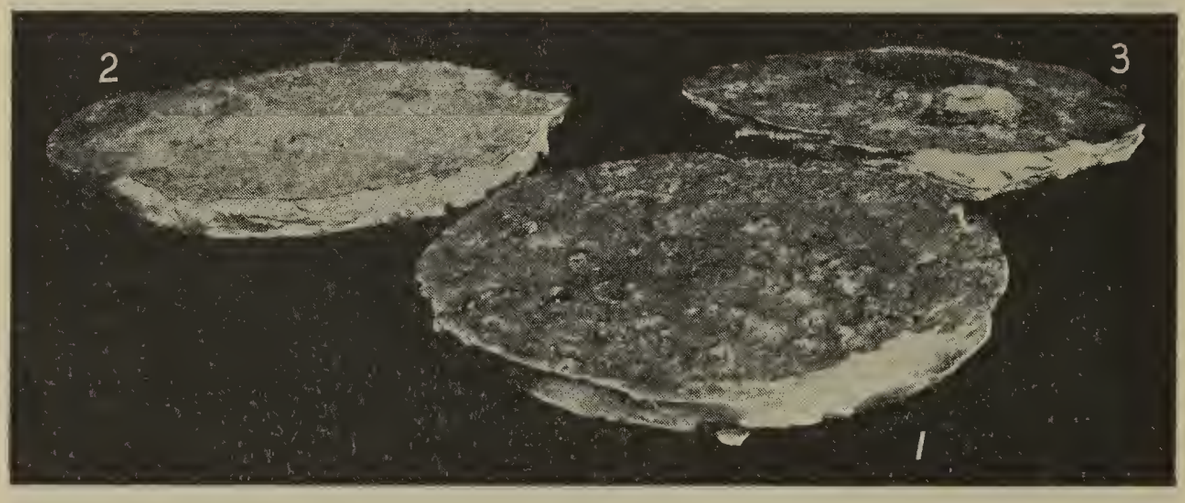


FIG. 3.

the gas company's tanks have been analyzed by Dr. Stillman, of Stevens Institute of Technology, Hoboken, N. J., who reports as follows:

THOS. B. STILLMAN, M.S. Ph.D.,
Chemical Engineer.

HOBOKEN, N. J., October 29, 1908.

ADOLPHUS A. KNUDSON,
Electrical Engineer,
New York City.

Dear Sir:—The sample of water from bottom of oil tank marked No. 11 contains 1.6 per cent. mineral residue—mostly salts of soda. The sample of water from bottom of oil tank marked No. 7 contains 1.3 per cent. mineral residue—mostly salts of soda.

Both solutions react alkaline.

Respectfully yours,

(Signed) THOS. B. STILLMAN.

The oil in Tank No. 7 has been standing about one year, that in No. 11 somewhat longer. This oil is believed to come from Texas.

The bottom of No. 11 was not examined, as it was in use.

ELECTRICAL CONDITIONS.

Tests have been made to ascertain the electrical conditions due to stray railway currents, as possible corrosion from this source, it was thought, might be going on at the *underside* of these bottoms. Railway currents have been discovered passing through the gas company's yard, mostly going through and leaving the ends of power house suction pipes placed in river. One of these suction pipes, a 20-inch cast iron, entered the river quite near these oil tanks. Current flow on this pipe was found passing to the river ranging from 1 to 4 amperes, at times 6 amperes, maximum. The strainers at the end of this pipe have twice been destroyed by electrolysis.

The voltmeter readings between tanks and river were taken several times during the past year. The tanks have always been found positive to the river. In March, 1908, 2.0 volts maximum was observed. A tank used for tar stands close to these two tanks and is under precisely the same electrical conditions as to stray railway currents, but no corrosion has developed at the bottom, so far as known, either inside or outside. Tests for current flow on the 6-inch pipe connecting with the two tanks, shown in drawing, has also been made several times during the past year, but in no case has a flow been detected. There being no other pipes connecting with the tanks, it was concluded that no current was leaving them, notwithstanding the voltmeter indications. Had there been corrosion upon the *exterior* bottoms as well as upon the interior, with the two forces at work, the life of these tanks would certainly have been much shorter.

THERMO ELECTRICITY.

Tests were also made for thermo electricity as a possible contributing agent for corrosion. A thermometer observation on a hot day in summer at Tank No. 7 at the interior bottom regis-

tered 27.5° Cen. At the interior top 39.5° Cen., a difference of 12° Cen. A millivoltmeter reading between the bottom and top 0.2 millivolt, bottom positive. Shady side of tank postive to sun side 0.15 millivolts. This tank was empty, but the same tests were made on one in use, resulting practically in the same readings, both temperature and electrical. The millivolt test on Tank No. 7 between top and bottom was repeated in winter, when all parts were cold, and with no result. It is a question if these indications of feeble thermo-electricity play any part in the corrosion discovered, but it is mentioned as an item for possible discussion.

Oil Tank No. 9 in one of the Standard Oil Works.

The pittings and holes being very similar to those discovered in tanks No. 7 and 8, it was not deemed necessary to obtain further casts. At one time it was thought by the manager that the cause of corrosion was due to a certain class of oil stored in the tank, but he found that tanks containing white oils were also similarly affected. In this tank the roof supports were of steel, of the umbrella pattern. A second pipe is used for drawing off the oil. This pipe is called a swing pipe. It has a loose joint at the base and a chain attached at the far end, the pipe being about 20 feet long; the other end of the chain passes through an opening in the roof. The end of the pipe is thus raised or lowered so as to take the oil from near the surface. When we visited this tank a few weeks ago it was cleaned out and one of the side plates removed preparatory to placing in it a new bottom, a contract having been let for that purpose, the former one being so badly corroded it was deemed unfit for further use. The position of the pittings and holes in this tank floor was very similar to those in tanks Nos. 7 and 8, as shown by the drawing, Fig. 1. This tank has been in use 13 years. The interior sides of all three of these tanks were carefully examined and no sign of corrosion of any kind could be found, in fact, the outline letters and figures on the interior of one of the plates in Tank No. 9, made with chalk or paint when the tank was made 13 years ago, could be made out, although both letters and figures were upside down as viewed from the floor.

DANGER FROM LIGHTNING.

If we may digress a little from the main subject, we would like to call attention to the great danger of explosion and fire in oil tanks from lightning by the presence of this chain on the swing pipe. In 1881 the writer called attention to this danger in a paper before the New York Electrical Society, on the subject of "Cause of Explosions in Oil Tanks," such explosions being quite frequent at that time. As an illustration, a small model of a tank was used, and a static machine to produce the electricity; through an opening in the side of the tank the chain could be seen. When discharges were made upon the roof of the tank representing lightning, sparks were observed at nearly every link of the chain inside. As an obvious remedy, we suggested at that time, to substitute a wire rope in place of the chain, which would be a continuous conductor, and if lightning came that way it would pass off silently with no sparks. A letter from the manager of a large oil works in Pennsylvania informed us that they had even gone further than our suggestion, and had not only taken off the chains, but left out the swing pipes in all new tanks that were built, using other means to draw off the oil. It may be, judging from the one discovered in the tank recently examined (No. 9) that there are others with chains in use at the present time, and those in charge of oil works have not realized the danger, and this point may possibly come to their notice. It was a surprise to the writer that something worse than a destroyed bottom has not happened to this tank. The importance of this point, as viewed by the writer, is the excuse for turning aside from the subject for a moment.

CAUSE OF CORROSION.

In this case of such pronounced corrosion in oil tanks, there must of necessity be definite and pronounced cause. In looking for such cause or causes, the following data has been obtained. It is well known that crude oils contain more or less sulphur. In the purifying process we find it customary to subject such oils to treatment with sulphuric acid, and after that, to caustic soda. The oil is then, after each treatment, washed with water. It is reasonable to suppose that traces of acid or alkali remain

with the oil when shipped away. The superintendent of an oil works has informed us that on one occasion 75 barrels of sediment, or "B. S." as it was called, was removed from one of his tanks, which, he said, had a large percentage of caustic soda. When oil is shipped by steamers or lighters on the sea coast, which is often the case, there is more or less leakage, and salt water is added to the mixture, also rain water leakage through the roofs of the tanks. This layer of water which rests upon the bottom of oil tanks contains electrolytes, which are the products of the chemicals referred to, and are distributed over the surface of the iron bottom. Either through impurities of the iron, or from other causes, potential differences obtain, in other words, innumerable small galvanic couples are originated, which, when started, carry on their work of destruction. These galvanic effects, as shown by the plaster casts, indicate growth; some are most minute, as though in infancy, others larger, while some appear to have reached maturity, as shown by the large pittings, resulting in holes through the metal in many cases.

A more satisfactory explanation of this cause of corrosion will doubtless be found in the following extracts of letters to the writer, from Prof. Allerton S. Cushman, author of two recent papers on the subject of "Corrosion of Iron," of which you are no doubt familiar. At Dr. Cushman's request, we furnished him with the data of these cases of oil tank corrosion, in which he was much interested, and he has kindly given his opinion, with permission to use same in this paper.

WASHINGTON, D. C., October 1, 1907.

I have read your letter with care, and I can only say that I am not surprised that the bottom of the oil tanks, in which you are interested, pitted as rapidly as they did. According to the modern theories of corrosion, the electrolysis is not caused so much by outside electrical currents or differences of potential as it is by local difference of potential set up within the metal itself, owing to concentration changes in the distribution of impurities . . . .

In your tanks, which undoubtedly contained at the bottom a layer of water, in which electrolytes of various kinds were dissolved, you had the ideal conditions for pitting to take place. In my opinion, a fairly liberal application of bichromate would protect the bottoms of such tanks as those in which you are interested.

He also states under date of October 1st, 1908:

Since the date of the letters you mention, however, my studies have taught me that the concentration changes in the distribution of the impurities in the iron is only one of the controlling factors in stimulating cor-

rosion. The physical condition of the surface is also important, the slightest identation, scratch or cut in the surface appears to become positive to the surrounding area and thus forms a nucleus for a future pit hole. It is probable that in the best practice hereafter attention will have to be paid not only to the method of manufacture of the steel as affecting its chemical constitution, but also to the condition of finish of the surface which is going into use.

Referring to the surface abrasion as a contributing cause for corrosion, it has been noticed by the writer there were many corroded rivet heads on the floors of all three tanks. Some partially, but many entirely eaten away. Dr. Cushman's remark as to indentation, scratches, etc., forming a nucleus for future pittings and holes may apply to rivet heads, as well as other parts of a tank bottom, as there is no lack of opportunity for surface abrasion at such points, either in the process of riveting, men walking over them, or other causes.

REMEDIES.

The use of bichromate solution as a protective measure for corrosion of iron has been strongly recommended by Dr. Cushman in his papers and letters, and to myself at a personal interview. His opinions are, it is well known, based upon the results of carefully conducted experiments and we believe worthy of the most favorable consideration. If this method is applied to oil tanks, we think it should be sent into the tanks from the same intakes as the oil, as in such case the remedy would be distributed where most damage occurs, as indicated by the shaded parts of drawing, Fig. 1.

In one instance, as a protection from corrosion, it was proposed by the manager of an oil works to place a layer of concrete upon the floor of tanks. We expressed doubts of this method being effective protection, in view of former experiments by the writer on corrosion of iron in concrete.*

Owing to the well known absorption qualities of concrete water containing electrolytes would naturally pass through it to the iron bottom, probably causing the concrete to crack at points where electrolytic action takes place. Attempts have been made by some to prevent the absorption properties of concrete by incorporating with it certain waterproof materials, but we have

* Transactions American Institute Electrical Engineers, Vol. XXVI, Part I, 1907.

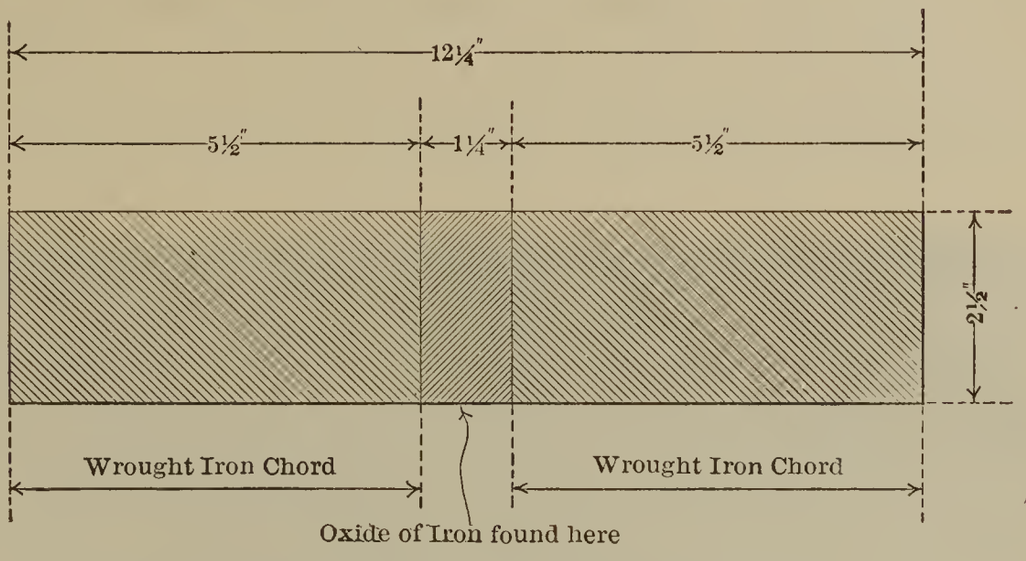
no data to present at this time of the efficiency or otherwise of concrete so treated.

CORROSION OF OTHER STRUCTURES.

Referring to the Rochester steel conduit, Mr. Richard H. Gaines has given an interesting account of the corrosion of this water main in a paper before this society at the Albany meeting, April 30, 1908, printed in the transactions. We have carefully read this account, and we are inclined to believe with those who took part in the discussion that the author's conclusions should not be accepted as final, where he states p. 87 (2), "The corrosion of the Rochester steel conduit was caused by electrolysis, the current for which resulted from chemical processes between water solution in the soil and the metal." While this may be true at some portions of the pipe, we notice that after a long account giving analysis of soils, and of composition of metal, character of coatings, etc., not one statement is made of the methods of measurements by the "electrician from the municipal laboratory," upon whose brief report that "no measurable current could be found with the millivoltmeter from outside sources," it is concluded by Mr. Gaines that no stray currents past or present should be considered as a cause for corrosion on this main. Under these circumstances, we do not think this a fair conclusion. Laboratory electricians are not always well equipped either with exeprience or instruments in locating or tracing stray currents. There are no details of the measurements, or if 24-hour readings were made, the latter to determine if the electrical conditions were the same during the entire 24 hours. It often occurs that the source of power upon railway lines are changed, operating from a sub-station during the day, and from another part of city or country at night, from a central station, when totally different results are obtained as to current flow on pipes. It is quite unusual to find pipes as badly corroded as this main is reported, where such corrosion is entirely due to "chemical processes."

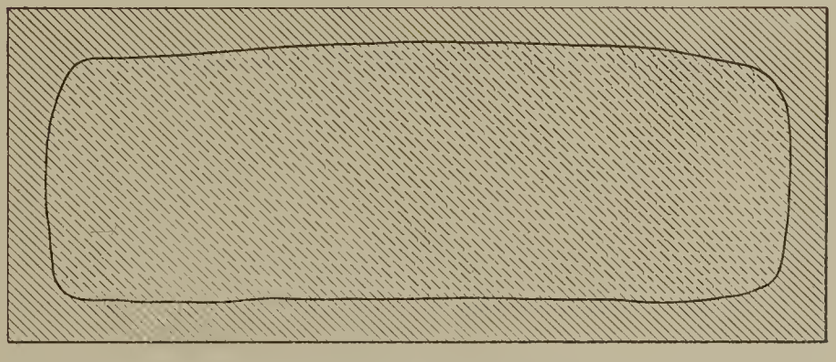
Another point has been noticed in underground electrolysis in an interesting paper before this society, April 30, 1908, by Prof. Burgess, also at the Albany meeting, entitled "Corrosion of Iron from the Electro-Chemical Standpoint," printed in the

transactions. In this paper corrosion of underground structures by straying railway currents are referred to, in which the author seems to question certain testimony that had been given by experts to the effect that a flow of current through the earth



Cross section with dimensions of two wrought iron bridge chords, before being damaged by electrolysis.
Point St. Bridge, Providence, R.I.

"A"



SECTION OF ONE OF THE WRO'T IRON CHORD MEMBERS OF POINT STREET BRIDGE

Original section of Chord before being affected with electrolysis.

Section of Chord after being affected with electrolysis.

Area of original section 12.375 Sqr. Inches. Area of present section 8.41 Sq'r. Inches. Percent of loss of section 32 Per cent. Area worked out with planimeter.

"B"

had resulted in the deposition of a coat of iron upon pebbles in the earth. I do not know who gave this testimony, but am sure they were correct, as such finds are not unusual.

I have here two pebbles of a number found so coated, which

were taken from the side of a cast iron pipe which had been practically destroyed by electrolysis from stray railway currents, in the city of Dayton, Ohio. These may possibly be some of the very same pebbles to which Prof. Burgess refers.

These and other samples are submitted for your inspection. The deposition of both iron and lead has often been found by the writer upon pebbles, stones and soils adjacent to pipes which were undergoing electrolysis. We have some samples here per-

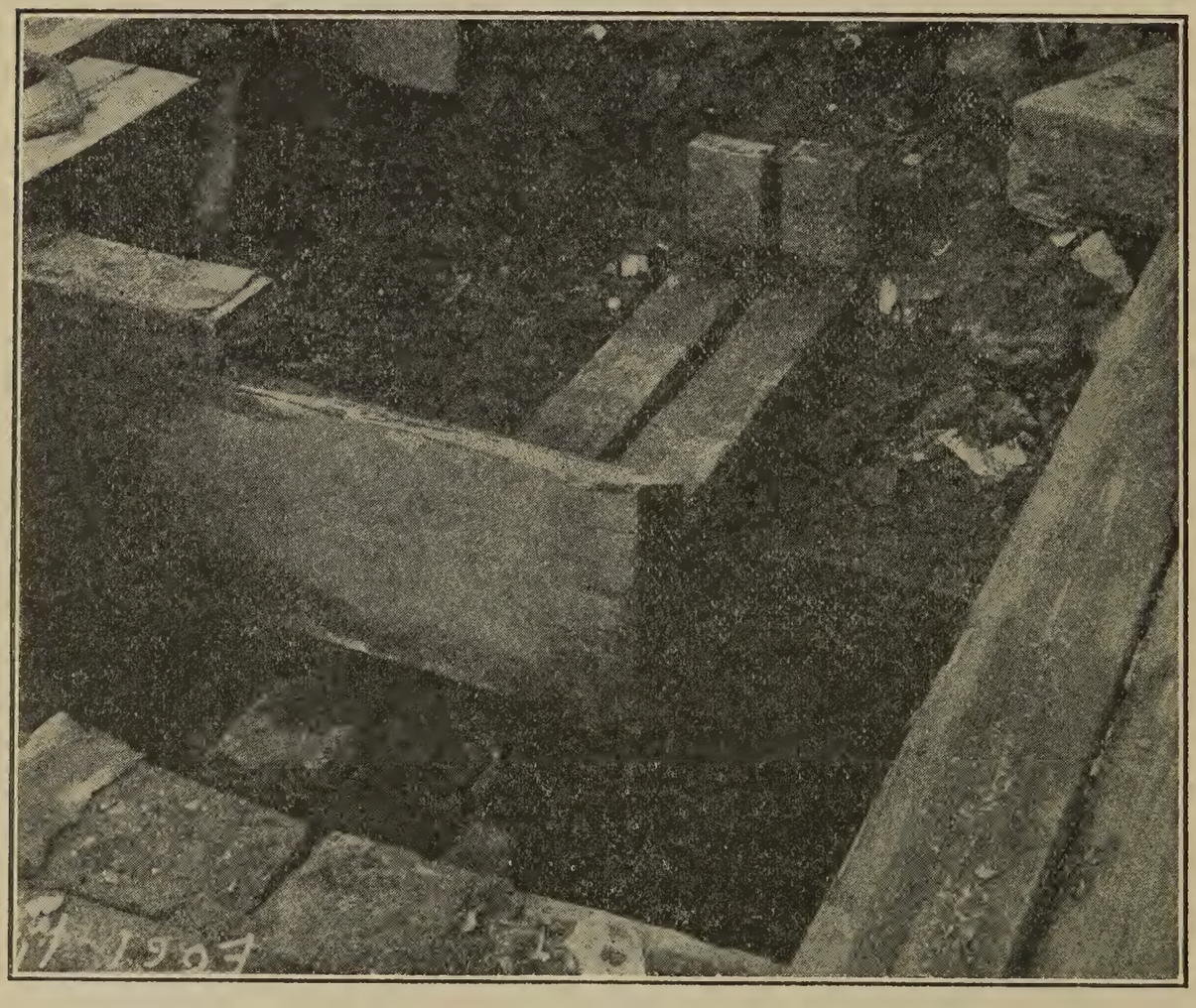


FIG. 4.

sonally obtained only yesterday, of stones and pebbles, which lay near a 12-inch steel water pipe which was delivering railway current into the soil, which show more or less of such deposit. This pipe, which is in Edgewater, N. J., is badly corroded. Next to the pipe was found a complete covering of black carbon of about ⅛-inch thick; at some points on the pipe there was a bright, metallic luster; next to the carbon distributed in the soil and upon stones was over an inch of iron deposit.

Samples of lead deposit are shown, also personally obtained from a lead service pipe in Cleveland, Ohio, which lay in sand. This pipe was so badly injured by electrolysis that about 25 feet had to be replaced with new pipe. These samples appear to be as heavy as an equal amount of lead.*

While it is appreciated that the electro deposition of iron is difficult under the most favorable circumstances, these samples so coated are shown as a suggestion that there may be something in the process of underground electrolysis not fully understood.

Another sample of corrosion of iron by *stray current* process was shown after adjournment, and this data obtained later. This sample is a mass of oxide of iron 5 inches long by 2 inches

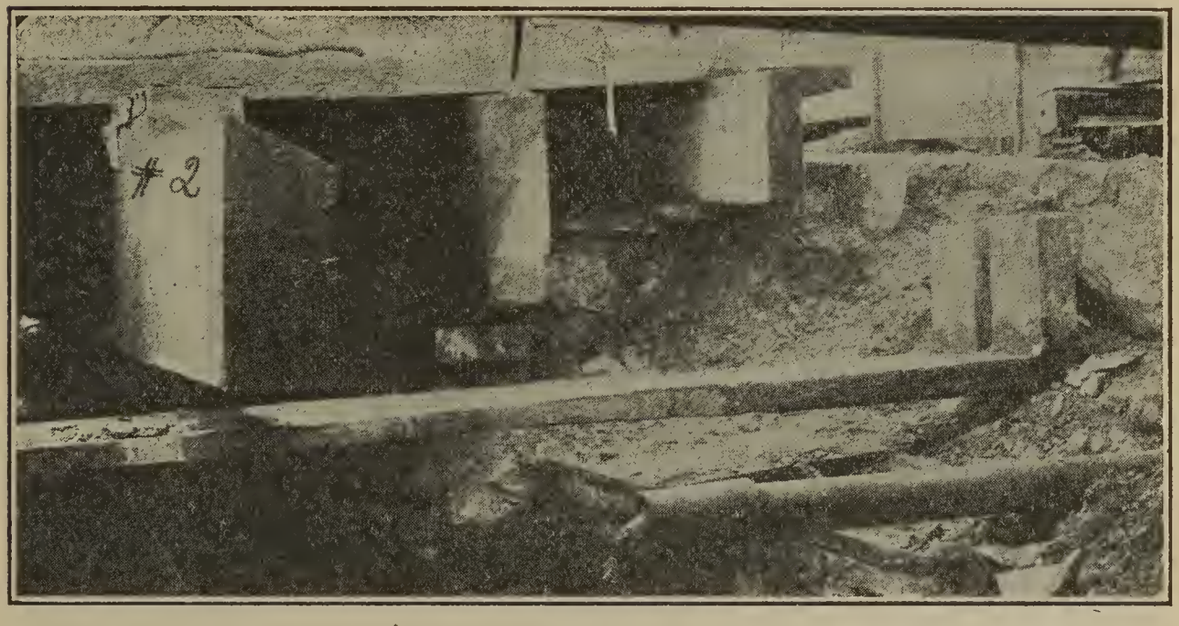


FIG. 5.

deep, and is 1⅛ inches to 1¼ inches thick. It was taken from between two bridge chords which lay 1¼ inches apart, from a bridge that was taken down and replaced by a new bridge, in the city of Providence, R. I. These bridge chords where they lay in soil were badly corroded, and when found were surrounded by a mass of oxide, the same as this sample. Figs. 4 and 5 are photographic reproductions of same; Fig. 6, corrosion at ends of spans. The dimensions of these chords, which were of wrought iron, are given in drawing "A." The comparison of damage with the original size is given

* After Prof. Burgess had inspected these samples he stated they were composed of white lead.

in drawing "B." Electrical measurements have been taken upon this bridge for some years past by the writer, who has often predicted damage. The bridge has always been found positive to the soil at the eastern end where this corrosion was discovered. In 1902 corrosion and holes were found upon one of the sheet iron trusses where it entered the soil. The railway power house was a short distance from this bridge on the west bank of the river. Submarine cables from the power house were attached to a cable box close to the eastern end of the bridge.

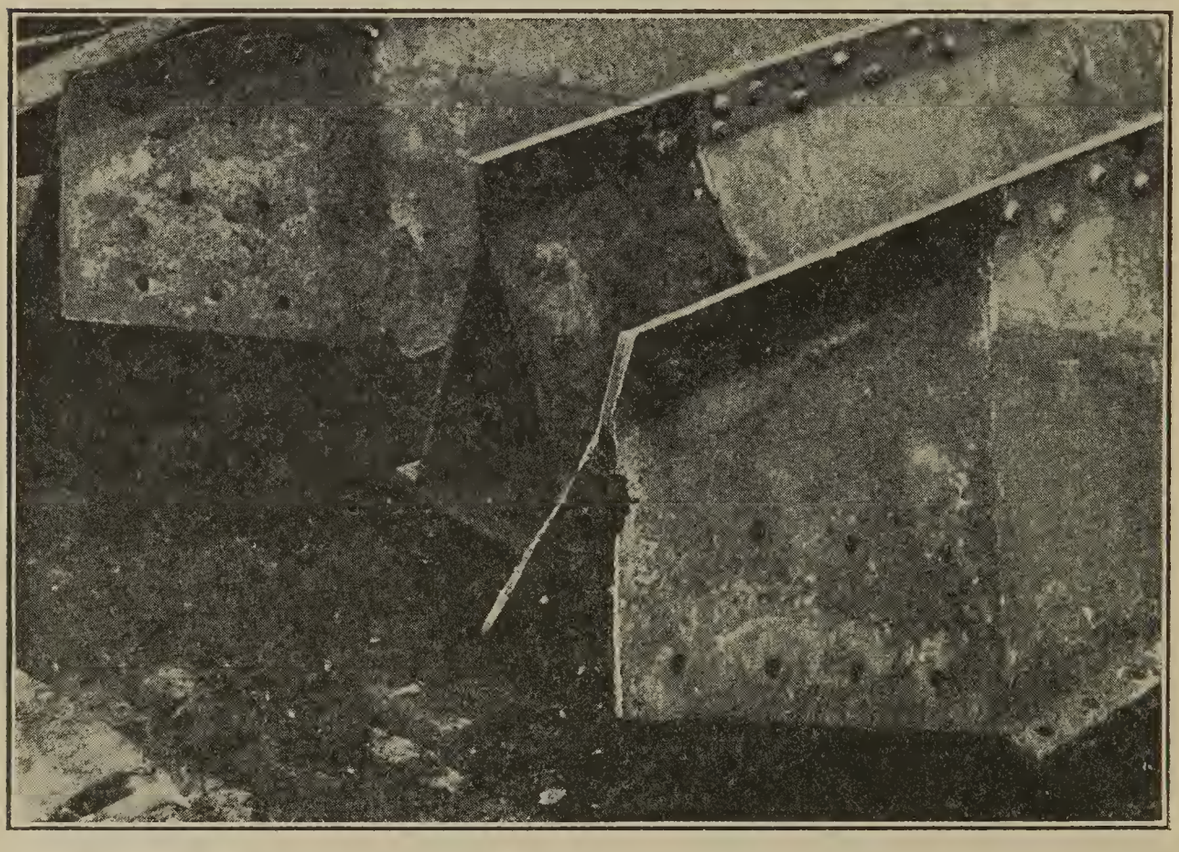


FIG. 6.

Stray currents have been found on this bridge from July, 1900, up to September, 1903, when they were checked by a change in the railway return conductors, which were placed on poles and the cables abandoned.

This sample and data is an example of what is possible in many bridges in this country.

In conclusion, from the standpoint of the electrical engineer, we are grateful for advice in the solving of electrolysis problems as to cause, effect and remedy in cases where they partly

or wholly refer to electrochemical action. On the other hand, personally, we are always willing to furnish information obtained through long practice as to methods for tracing, locating or identifying stray currents causing electrolysis. It would seem, therefore, as Prof. Ganz has stated, that co-operation in important cases between the stray current specialist and the electrochemist would be desirable, and to that end we have brought this case of oil tank corrosion before this society for discussion, as well as other matters, believing they are of the widest importance, and particularly refer to electrochemistry.

DISCUSSION.

MR. S. S. SADTLER: I had a little experience with oil tanks, and the main feature that came to my notice was that the soil may be quite acid from the sludge of the oil, and it seems to me that a great deal of the corrosion of the bottom of the tanks may be the chemical action of the acid in the soil. It may be electrolytic, notwithstanding, but it is not from any outside source, whereas in the case of piping or pipe lines, it is different.

MR. ALLERTON L. CUSHMAN: I will point out that a solution of an electrolyte in contact with the surface of iron and steel, if it is slightly alkaline, that is if the concentration of the hydroxyl iron has not reached a certain point, the corrosion seems to be stimulated, probably owing to the fact that the chemical action of the electrolyte is seggregated, so to speak. That was plainly brought out first in the paper by an Englishman named Krubb, who in the interest of a water company made some exhaustive tests of the effects of different waters on the surfaces of different kinds of steel. He found that the weakly alkaline waters corroded and pitted his samples more than the neutral water did, although not more than the acid water did. He pointed out that the pitting effect appeared to be worse where there was a weak alkaline reaction. In this case, if the reaction was weakly alkaline, it would have just that stimulating effect, that Mr. Knudson observed.

MR. C. F. BURGESS: The author of the paper just presented made the statement, if I understood him correctly, that iron could be deposited upon pebbles, and he gives some statements to illustrate that phenomenon. Did I understand you correctly?

MR. A. A. KNUDSON: Yes, that is correct.

MR. BURGESS: It is very surprising to me, to be told so authoritatively that this can be done. I am unconvinced, however, and the specimen shown does not completely prove the assertion. These pebbles certainly have a coating of iron in the form of compound, but not as a metallic iron. It may be a hydrate, or an oxide, but I do not think they are coatings of metallic iron. It may be possible that the coatings oxidized since taken out of the earth, but I should like to be more firmly convinced of the possibility of doing something which you cannot do in the laboratory—that is, to plate iron on a non-conducting surface. Any surface has to be made conducting, even when coating with copper and other metals, which are very easily deposited, before you can get a metallic coating.

MR. W. H. WALKER: I think a possible explanation for the appearance of the iron upon the pebbles mentioned is, that what Mr. Knudson terms iron is really oxide of iron, and deposits on the pebbles through the migration of the soluble ferrous iron from the pipe through electrolysis, and subsequent precipitates upon the pebbles as ferric-oxide through the agency of atmospheric oxidation. There is no metallic iron on the pebbles, but only iron oxide.

PROF. J. W. RICHARDS: The deposit on the pebbles is not magnetic, so it is not iron, at the present time.

MR. KNUDSON *(communicated)*:

UNITED STATES DEPARTMENT OF AGRICULTURE.
OFFICE OF PUBLIC ROADS,
WASHINGTON, D. C.

November 28, 1908.

MR. A. A. KNUDSON,
50 Pine Street,
New York City.

Dear Sir:—I enclose you herewith some results obtained with the samples of water sent by you from the bottom of your oil tanks.

You will see that the corrosion of both iron and steel proceeded very much more rapidly in the alkaline electrolyte than it did in the ordinary laboratory distilled water. We have found that by increasing the alkalinity of the water to about three times its present strength the corrosive action was entirely stopped. I did not have sufficient quantity of your

water to determine this exactly or to my satisfaction. But I feel sure that if you will add a certain amount of sodium hydrate to the oil tanks, thereby keeping the water which collects in the bottom sufficiently alkaline, you will have no further trouble with the rusting out and pitting of the bottom plates.

Very truly yours,

(Signed) A. S. CUSHMAN,
Assistant Director.

UNITED STATES DEPARTMENT OF AGRICULTURE.
OFFICE OF PUBLIC ROADS,
WASHINGTON, D. C.

November 28, 1908.

Water from tanks of New Haven Gas-Light Co.
Water from bottom of tank No. 7, taken October 16, 1908.

Non-volatile solids.....962.8 parts per million.
Alkalinity equivalent to.....673.9 parts KOH per million.

Four pieces of steel and four of iron tested with air in above water for 36 hours gave following losses:

Iron.	*Steel.*
.0667	.0887
.0560	.0810
.0717	.0968
.0614	.0922
.2558	.3587

Similar pieces of steel and iron treated the same way in distilled water gave following losses:

Iron.	*Steel.*
.0415	.0612
.0326	.0648
.0365	.0602
.0469	.0542
.1575	,2404

Water from oil tank No. 11, taken October 20, 1908.
Total non-volatile solids.....1399 parts per million.
Alkalinity equivalent to..... 921 parts KOH per million.

MR. W. S. LANDIS *(communicated):* In regard to the sample of "oxide of iron" taken from between the chords of the Providence Bridge I find it to be composed of:

Iron disulphide	85.19
Hydrated iron oxide.....	10.56
Undetermined (SiO_2, C, CaO, etc).....	4.25
	100.00

The sample was about the size of a fist, brittle and much resembled a decomposed pyrite.

MR. KNUDSON: The term as used in Prof. Burgess' paper, "The Deposition of a Coat of Iron," was understood to mean either iron in a metallic state or as ferric-oxide. All the samples presented show the coating of oxide.

In some of them, at places where scraped with a knife a metallic luster is observed indicating the metal; these, however, will not attract a delicately poised magnetic needle.

In the case of the cake of oxide removed from between bridge chords, the needle is attracted at a few points on its surface only. It is our belief, therefore, that these deposits or coatings on stones, and pebbles, are almost entirely iron in the ferric state and in some rare cases, small particles of metallic iron can be found.

A paper read at the Fourteenth General Meeting of the American Electrochemical Society in New York City, October 30, 1908; President E. G. Acheson in the Chair.

THE PROTECTION OF STEEL AGAINST CORROSION.

By Maximilian Toch.

The extensive research work done in investigating the underlying causes of the rusting of iron has given rise to some theories, notably the carbonic acid, the peroxide, and the electrolytic, in support of which many arguments and experimental evidence have been advanced. The important place which iron and steel occupy in the modern industrial field makes their protection against decay a matter of particular interest.

That carbonic acid will aid and accelerate corrosion is evident, but that its presence is essential before rusting can take place has been clearly disproved by the work of Whitney, Cushman and Walker. Dunstan, Jowett and Goulding have not been able to prove experimentally the existence of hydrogen peroxide as a transition product in the corrosive process. It has also been demonstrated that iron is not acted on by a pure neutral solution of hydrogen peroxide; although rusting would take place if ordinary peroxide was used, since this contains acid and other impurities. The electrolytic theory explains the corrosion of pure iron in pure water by the interchange of dissociated hydrogen with iron, with the liberation of the hydrogen and the solution of the iron as ferrous hydroxide.

W. R. Whitney (J. Amer. Soc., Vol. 25, No. 4, April, 1903) expresses this as follows: "Hydrogen acts as a metal and is electrolytically classified in the group with copper when compared with iron and zinc. That is, if a cell were made on the Daniell model, iron being used instead of zinc and hydrogen in place of copper, the cell would generate a current when the iron and hydrogen electrodes were connected. Iron would then dissolve with a velocity dependent on the total resistance of the circuit. So, also, and for the same reason, iron when placed in a solution

containing hydrogen ions will dissolve as the hydrogen precipitates, just exactly as when placed in a copper salt solution. That iron does oxidize or dissolve in all solutions containing appreciable quantities of hydrogen ions is well known. The electrochemical relationship between iron and hydrogen is the primal cause of rusting."

When the technical side of the question is considered we have to deal with not only hydrogen ions due to the dissociation of water as causing electrolysis, but also electrolytic reactions between the iron and the impurities or small percentages of other metals which are added or are naturally present in the material and give it desired properties.

A. S. Cushman (Bul. No. 30 of U. S. Dept. of Agr., July, 1907) thus describes the action which takes place when a sheet of steel or iron corrodes: "If a section of rolled metal, such as sheet or plate is immersed in water, if the electrolytic theory is correct, rusting must take place with the establishment of positive and negative spots or areas. As the positive points iron will pass into solution and be rapidly oxidized to the loose colloidal form of ferric hydroxide which is characteristic of rust formed under these conditions. It is a well known fact that colloidal ferric hydroxide will move or migrate to the negative pole if subjected to electrolysis. We thus will obtain two separate effects, the one in which a positive center is surrounded by a negative area, and vice versa. In the first instance the ferrix hydroxide would be piled up in a crate formation while the metal is eaten out at the center. In the latter the effect would be reversed, and the metal would be attacked in the surrounding area and hydroxide piled up in a cone at the center. That this is precisely what takes place whenever a sheet of metal rusts under water a low power microscope very clearly shows."

The work of different experimenters has shown that solutions of various substances have the power of rendering iron passive for variable periods, the pacifying agents being alkalies, chromates and chromic acid, nitric acid, potassium cyanide and other substances of which, however, the alkalies and chromates are the only ones supposed to be of practical value. I made experiments with a number of rust inhibiting substances, using samples of steel obtained through the courtesy of the Union Pacific Rail-

road Company and the American Bridge Co. (U. S. Steel Corp.). Special thanks are due to Mr. E. H. Harriman, Mr. Gerber and Mr. Millar for these samples.

ANALYSES OF STEELS.

	Carbon	Silicon	Sulphur	Phosphorous	Manganese
1. Rail steel	.632%	.066%	.025%	.108%	1.10%
2. Bridge steel.....	.319%	.016%	.022%	.019%	.563%
3. Structural steel.....	.110%	.029%	.056%	.018%	.340%
4. Car steel	.289%	.070%	.024%	.002%	.440%
5. Rail sections and drillings	.550%	.100%	.040%	.090%	1.02%

These specimens were pickled in different solutions of various strengths for periods of from one hour to two days. The pacifying effect was a transitory one, lasting at most for several days. The object of these experiments was to determine whether it was possible to subject iron and steel building materials to some pickling process so that the passive state would continue for protracted periods. To be of utility, such a process must be quick and give fairly lasting effects. Pieces of steel were treated with chromic acid, calcium chromate, and potassium bichromate, using solutions of strengths varying from two-tenths to twenty per cent. As an instance, a piece of steel rail which stood in five per cent. potassium bichromate solution for eighteen hours, on being rinsed off and allowed to lie, within one hour showed the effects of corrosion in scattered spots distributed over the whole surface. Specimens of rail, bridge, car and structural steel were subjected to pickling in these solutions, with results which show that structural material treated in this manner would not be proof against the effects of a shower of rain or standing in moist air, more particularly when we take under consideration the gases and vapors found in the air in factory and manufacturing districts.

Steel plate in chromic acid 20% sol. for 6 hrs.—corrosion in 2 hours.

Steel strip in 7% potassium bichromate for 2 days—corrosion in 2 hours.

Both samples shown in ferroxyl reagent.

On trying samples of steel and iron in ferroxyl reagent made according to A. S. Cushman and Wm. H. Walker, I likewise found that corrosion took place with the formation of changing

positive and negative areas. However, when two pieces were allowed to overlap or touch, the blue coloration always developed, showing that iron went into solution and rusting took place. The formation of electro positive and consequently corroding areas invariably occurred at the place of contact between the pieces of metal. Thus, in a steel or iron structure, although rusting might take place along the whole surface of a girder, beam or plate, it would be much more active at the points where the pieces are bolted or riveted together. Since the passivity of iron cannot be made a fairly lasting property, other means must be used to protect metal structures.

It is universally admitted that the presence of water is necessary before corrosion can take place, since this is the medium which contains or supplies the hydrogen ions which are necessary for interchange with the iron. Thus, in order to prevent rusting, you must either exclude water entirely, or have some substance present which will prevent the formation of the hydrogen ions. The only way in which this problem can be solved is by the application of a paint which will either protect the surface by being absolutely impervious to water and therefore resistant to the hydrolizing action, or one which contains in itself either as a part of the vehicle or pigment some substance which can produce the passive state.

A little over a year ago I made some samples of anti-corrosive paints in which chromates were used. These samples were painted out on strips of steel and exposed to the weather. From time to time the samples were examined by removing a little of the paint, and after exposure for one year the surface of the steel has been found exactly like those samples which were coated with good paints only.

In a large line of experiments pieces of steel were first coated with various soluble chromates and then coated with linseed oil paints and bituminous protective paints. Some pieces of steel were coated with paints without the application of any inhibiting solutions, and on exposure to the elements it was found that at the end of a year the protective effect in all the samples was practically alike, depending largely upon the material itself, as, for instance, ordinary graphite paint, on account of its thinness of film, did not protect as thoroughly as either oxide of iron or

red lead, for the obvious reason that oxide of iron and read lead produced thicker films which withstood the abrading action of the elements.

Another series of experiments were made in which ammonia chloride was added to the ferroxyl reagent. The addition of ammonium chloride hastened oxidation, as is well known, and it was again found when placing the strips in a medium of gelatine containing ferroxyl ammonium chloride, the chromates retarded rusting, in some instances to some extent; but in the end, little, if any, difference was noted between the samples which were dipped in the inhibiting solutions and those which were plain.

It must be understood that this treatise is not intended by any means as a criticism of the excellent work done by Cushman and Walker, but simply deals with the plain fact that steel used in construction is handled very roughly between the mill and the place of erection, and wherever abrasions occur, rust is sure to follow, whether an inhibiting medium has been used or not. The whole point which I wish to make is that up to now no good medium has been devised for inhibiting corrosion of iron or steel in transit, for the removal of the inhibiting material means the removal of the pacifying agent, and it might be well to try the experiment of sending structural steel without any coating on it whatever, then using a soluble or semi-soluble chromate, such as a mixture of calcium or zinc and potassium chromate, applying these materials after the steel is in place, then coating with the usual protective paints. An experiment of this kind on a stand pipe, steel stack or similar construction which is not intended to be enclosed in masonry, might show some desirable results. The results of the experiments made by the author can be summed up as follows:

That after a lapse of six months there is no difference whatever between the steel that has been coated with a good protective coat and one that has been pacified and painted with a similar coat of paint; that the greatest danger lies in the zone where two pieces of steel come in contact, and that at such places an alkaline paint or an electrical insulating paint should be placed, and at such other points where two pieces impinge or come in contact.

Where rust has already started, the chromates have no effect,

for it appears that the electrochemical phenomenon progresses just the same as if no inhibiting medium were used.

It is gratifying to me to be able to acknowledge publicly the work which my assistant, Mr. V. P. Krauss, rendered in performing faithfully the experiments alluded to.

DISCUSSION.

PROFESSOR GANZ: I should like to ask Mr. Toch whether he considers it necessary in the case of a pipe which has been in the ground for some time to first coat the pipe with some kind of paint before imbedding it in asphaltum, as I have suggested?

MR. TOCH: Do you refer to a gas pipe?

PROFESSOR GANZ: No sir.

MR. TOCH: What kind of a pipe?

PROFESSOR GANZ: Wrought iron pipe for special purpose.

MR. TOCH: It will not be necessary to paint it with oil paint. It would be better to apply an insulating paint, hydrocarbon paint, and instead of pouring pitch around the pipe, pour half neat cement. This would be just as cheap, and far better.

MR. ALLERTON L. CUSHMAN: In regard to Mr. Toch's experiment with the inhibition paint compound, Mr. Toch, I suppose painted this iron, and did not then cover that with the best excluding compound that was available. The object of the inhibitor is to keep in contact with the surface of the steel a very slightly soluble chromate, so that if any water does get through the excluding paint surface, it will also have to go through the inhibiting surface, and when it reaches the surface of the steel, the water must contain in solution some chromate of iron. The question is will it work out practically? Theoretically the idea seems to be all right.

I and my colleagues on a special committee appointed by the American Society for Testing Materials investigated this subject, and our desire has been to be extremely conservative and never to state that this was an accomplished fact, but that it was an interesting thing, and well worth practical investigation, and these investigations are now in process of being carried out. I am not surprised that in coating small samples of steel, with in-

hibitive pigments mixed in linseed oil, Mr. Toch found these compounds failed, although they were successful for a short time. That is precisely what I should have predicted would take place, because being compounds, if they are not protected they will naturally leach or wash out of the oil film in time, and the destruction of the steel will go on as usual. What is the object of carrying on this test, if there is already an exclusive compound of any sort in existence which will protect iron and steel? The universal testimony of engineers who have charge of large steel structures is that no such successful compound has ever appeared. If it had, I and my colleagues in this work certainly would not waste our time trying some others. As long as there is no such thing as a perfectly successful paint, we consider that this is a legitimate field for experimentation, but we do not desire to have anything prematurely said about it for we as yet have absolutely no data in hand.

MR. TOCH: In replying to Dr. Cushman, I wish to say that I naturally was very fair towards all samples and treated them all alike, and coated those with protective coatings which had the inhibitive material on them. In actual practice, when a beam or a billet is coated at the mill and carried fifteen miles, or fifteen hundred miles, as is sometimes the case, and great chains are wrapped around it to hold it on railroad cars, and men are constantly walking over the beams, no protective coating of .001 of an inch has been made that will withstand these attacks. In my experiments, I duplicated this state of affairs by scratching these plates, and tried to duplicate the conditions as nearly as possible.

I did not mean to imply that any material was in existence which was perfect, when I spoke of red lead. I mean to say that none of the experiments made with the inhibitive material where the inhibitive material was used and then painted, showed any better than the experiments where no inhibitive material and red lead and other good paints were used. That was all I intended to say. I wish to further say that I do not want to detract to the smallest extent in anything I said from the excellent work which is being done by Dr. Cushman and his associates. I simply wished to say that in my experience I did not find that the use of passifying materials was of any value before paint was applied.

Paper read at the Fourteenth General Meeting of the American Electrochemical Society, New York City, October 31, 1908; President E. G. Acheson in the Chair.

HEAT CONDUCTANCES THROUGH WALLS OF FURNACES.

By Carl Hering.

In calculating the loss of heat through the walls of furnaces, the cross section of different parts of the path of the heat vary very greatly, as the walls have a small surface on the hot side and a large one on the outside. For all but large, relatively thin walled furnaces, this variation in cross section is very great. To use the ordinary average cross section, that is, the arithmetical mean, as is usually done, it is not rigidly correct. In electrical furnaces in which the heat is expensive, its loss by leakage is of more importance than in fuel furnaces, hence it is desirable to be able to make calculations as accurately as possible, so as to prevent the loss as much as possible by good design and the best proportions.

The purpose of this paper is to give the rigidly correct results, to deduce important and useful relations from them, and to compare the correct results with those obtained from the approximate formulas in which the simple arithmetic mean is used.

The correct formulas are not easily found in books, and they are too tedious to use for the sometimes very numerous trial calculations necessary before the desired results are obtained. The error in using the approximate formula varies greatly, being sometimes negligible and at other times very large, amounting to several hundred per cent. Hence a comparison of the results obtained from the correct and the approximate formulas, is of importance.

Such a comparison in parallel columns and by means of a pair of curves, has the following advantages:

It shows the amount of the error in using the simpler approximate formula in any particular case, and therefore enables one to see whether it is appreciable or not.

It enables the results to be calculated by means of the simple formula, and then corrected by the given amount.

It enables the correct results to be found directly from the tables and curves without calculation, for the ranges given.

It may enable better approximate formulas to be determined.

The rigidly correct results enable instructive and valuable deductions to be made, which it would be very unsafe and even dangerous to make with the approximate formulas. Some of these deductions are quite surprising. They give one a better insight into the heat conduction through thick walls surrounding relatively small spaces.

The correct results also suggest a simple method by which the conductivity k can be determined under the same conditions under which it is to be used for calculations; for by measuring the real loss under given conditions, and knowing the correct formula, the true value of k may be readily calculated.

The correct curves also show graphically to what extent it is worth while increasing the thickness of the walls, because the thicker they are made, the greater will be the cross section of the outside layers, hence the less will be their insulating property per inch of thickness.

The well known formula for calculating the conduction of heat through bodies is

$$H = \frac{S}{l} kt \qquad (1)$$

in which H represents the gram calories per second which pass through from one side to the opposite one, if no heat is lost on the way; S is the cross section of the body in square centimeters, l its length in centimeters, k the conductivity found in tables which signifies the calories per second which will pass from one side of a cubic centimeter of the specific material, to the other, for one degree Centigrade difference of temperature, there being no loss of heat through the other four sides of the cube; t is the difference of temperature between the two faces, in Centigrade degrees.

Of all these quantities, S and l are purely geometric, that is, they are mere dimensions and have nothing to do with any physical constants; they are constants for a body of any given shape

and are independent of the temperature, the material or any other such factors. Hence they may be considered by themselves, and the relations between them will always be the same for all bodies of like size and shape. The present paper deals with this relation of S and l in such shaped bodies as constitute the walls of furnaces. The difficulty in determining the loss of heat through irregularly shaped bodies like frustrums, lies in this relation of S to l, and when this has been determined, the rest of the calculation becomes very simple.

This quantity S/l in the above formula signifies the conductance of any particular body of a given shape and size, for a conductivity equal to unity. It might be called a conductance factor. When thus considered it can be calculated for a body of any particular shape, and then is that number which, when multiplied

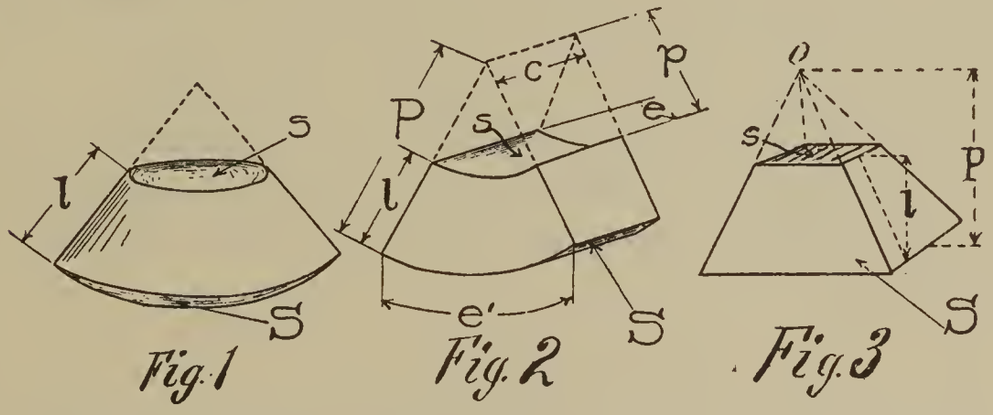


FIG. 1.—FRUSTRUM OF HOLLOW SPHERE. FIG. 2.—FRUSTRUM OF CYLINDER. FIG. 3.—FLAT FRUSTRUM.

by the conductivity, gives the total conductance of that body; and when this conductance is again multiplied by the difference of temperature, it gives the flow of heat through the body. In this paper the true values of this conductance factor have been calculated for numerous ranges of thicknesses in several typical cases.

It may be claimed that the value of k is so uncertain, that there is no need of accuracy in the other factors of the formula. The errors made by the usual way in which this conductance factor is calculated, are however sometimes very great; moreover if there are several uncertain factors in a formula, the results will be more reliable the less the inaccuracies and uncertainties involved in it, and it is hoped that with the great need of more accurate values of k, they will before long be forthcoming.

For the purposes of these deductions, furnaces are best divided

into three typical cases, spherical, cylindrical and cubical. Others must be considered to belong to the nearest typical form, or calculated by themselves.

Fig. 1 shows a frustrum of a hollow sphere, Fig. 2, a wedge shaped frustrum of a hollow cylinder, and Fig. 3 a flat frustrum whose two parallel sides are perpendicular to its axis and whose cross section varies proportionately with the square of the axial length from the center O; no other conditions are imposed and it may therefore have any shape of cross section, round, rectangular, or irregular. In all of them the resistance or conductance between them or thickness of the wall, being represented by l. The smaller or hot surface is s, the larger or cooler S.

To obtain rigidly correct formulas recourse may be had to the corresponding electrical formulas, and it is assumed that as far as these purely geometric relations are concerned, these formulas represent general physical laws, applicable to forces in general, hence to heat flow as well as to the flow of electricity.

From the wealth of information in the unpublished work of Dr. E. F. Northrup, the formulas for the electrical resistances of these three typical forms of frustrums are, for a frustrum of a hollow sphere:

$$R = \frac{rl}{\sqrt{Ss}}; \qquad (2)$$

for a whole cylindrical shell with open ends:

$$R = \frac{r}{2\pi c} \times \text{nat. log.} \frac{P}{p}; \qquad (3)$$

and for a pyramidal frustrum as above defined:

$$R = \frac{r}{\sqrt{n}} \left(\frac{l}{\sqrt{s}} - \frac{l}{\sqrt{S}} \right) \qquad (4)$$

The correctness of these formulas, as given here, is assumed.

The corresponding approximate but much simpler formula with which these are to be compared, in which the arithmetic mean is taken of the two surfaces S and s is

$$R = \frac{rl}{(S + s) \div 2} = \frac{2rl}{S + s} \qquad (5)$$

In all of these, R is the resistance in ohms between the two surfaces S and s stated in square centimeters, l is the length or normal distance between the two surfaces in centimeters, r is the resistivity in ohm-cubic-centimeter units, and in the case of the cylindrical one, P is the outer, p the inner radius in centimeters, and c the axial length of this cylinder in centimeters; n is the rate of flare in Fig. 3, such that $S = nP^2$ in which P is the perpendicular distance from the center O to the surface S. All these dimensional letters are shown in the diagrams.

In order to make direct comparisons, all these formulas must be reduced to the same terms; in practice, the quantities which are always known are the surfaces S and s, and the distance between them, or thickness of wall l; hence the formulas should all be reduced to these common terms.

The formula for the cylinder is not in a form suitable for comparison with the others, as it is for the whole cylindrical shell and not for a portion of it. To reduce it to a fractional part of the circumference, and to an expression which involves only the quantities S, s and l, like others, let e be the circular length of the curved inner edge of the wedge shaped cylindrical frustrum, as indicated in the diagram; then the resistance of this fractional part of the complete cylindrical shell will be $2\pi P/e$ times that of the whole shell; hence the resistance of the frustrum will be

$$R = \frac{2\pi P}{e} \times \frac{r}{2\pi c} + \text{nat. log } \frac{P}{p}$$

To reduce this to the terms S, s and l, substitute S/s for P/p, s for the surface ce, and for p its value $sl/(S-s)$ which is obtained from the relations $l = P-p$ and $P/p = S/s$; then the resistance of the frustrum becomes

$$R = \frac{rl}{S-s} \times \text{nat. log } \frac{S}{s} \qquad (6)$$

For the cubical frustrum Fig. 3, $2l = \sqrt{B} - \sqrt{A}$, and $S = 4P^2$, hence $n = 4$, and after making some simple algebraic reductions, equation (4) becomes

$$R = \frac{rl}{\sqrt{Ss}} \qquad (2)$$

which it will be seen is the same as equation (2) above for a spherical frustrum. Hence the comparison is reduced to only two forms, the spherical or cubical, and the cylindrical. It will be noticed that in the formula for the cubical and spherical frustrums the correct effective cross section is the geometric mean between the two extremes.

All these formulas apply equally well to either frustrums or to the whole of a complete body.

As the natural or Naperian logarithms are not so convenient to use, especially for fractions which may be near to unity, formula (6) may be written

$$R = \frac{2.3026\, rl}{S\text{-}s} \times \text{com. log } \frac{S}{s} \qquad (7)$$

These formulas all apply equally well to heat resistance, if r is the heat resistivity, that is, the reciprocal of the heat conductivity stated in C. G. S. units (gram calories per second for one cubic centimeter and one degree Centigrade), and R is the total heat resistance in the same units. Then the heat passing through such a body in gram calories per second is, analogously to Ohms law

$$H = \frac{t}{R} \qquad (8)$$

in which t is the difference of temperature between the two surfaces.

With the above formulas the heat lost through the walls of spherical, cylindrical and rectangular furnaces can therefore be determined directly. But they are somewhat tedious to use, when numerious trial calculations have to be made, particularly the one for cylinders. The approximate formula (5) is therefore more generally used.

It must be clearly understood that formulas (1) and (8) and any deductions made from them, refer to the temperatures of the heat conductor at its two end surfaces and have no reference to the surface contact resistances between the heating or the cooling gases or liquids; these must of course be added if they are appreciable.

It must also be understood that the conductivity of most heat insulators varies with the temperature, sometimes very decidedly.

In the formulas here given it is assumed that the conductivity is the mean for the particular range of temperature of any given case.

The present investigation however deals primarily only with the question of what shall be used as the mean or average cross section in formula (1) for such frustrums, or as the result cannot be given in just that form, it deals with the part S/l in that formula, and therefore is not concerned with either the conductivity or the temperature. It deals with the conductance for a conductivity of unity; it is like the calculation of the electrical resistance of an odd shaped body for a resistivity of unity, therefore independently of the material; the actual resistance being then readily determined by multiplying by the resistivity of the particular material used.

In what follows, conductances will be used instead of resistances, as the former are more generally used in calculations of heat; hence the results apply to formula (1) rather than (8), although in the opinion of the writer, when the walls of furnaces are composite, it is much simpler to use resistances instead of conductances.*

The following tables were calculated and curves drawn, for the three typical forms, spheres, cubes, and cylinders, from both the correct and approximate formulas, and for the range of thickness likely to be used in practice. They will be referred to below more specifically. In general, and for the purpose of making comparisons and reducing the number of tables and curves, the inside diameter of the sphere or cylinder, or the inside edge of the rectangular furnace, has been made the unit of comparison, and the thickness of wall, l, has been given in terms of this diameter; thus $l = 1$ means that the thickness is equal to the diameter. The thicknesses l in the tables and curves must therefore be multiplied by the diameters in any particular case to reduce them to actual inches or centimeters.

The tables give the two results in parallel columns, so that the differences are readily seen. The ratio between them is also given, so that the results may be calculated by means of the simple formula and then multiplied by the correction factor, if it is appreciable.

*See "Heat Conductance and Resistance of Composite Bodies." Hering. Electrochem. and Metallurgical Industry, Jan., 1909, p. 11.

The figures in the tables have been determined in such units that they may be used directly, thus saving all or nearly all calculation, when the thickness of the particular wall corresponds with one of the numbers given. This will be explained more specifically below.

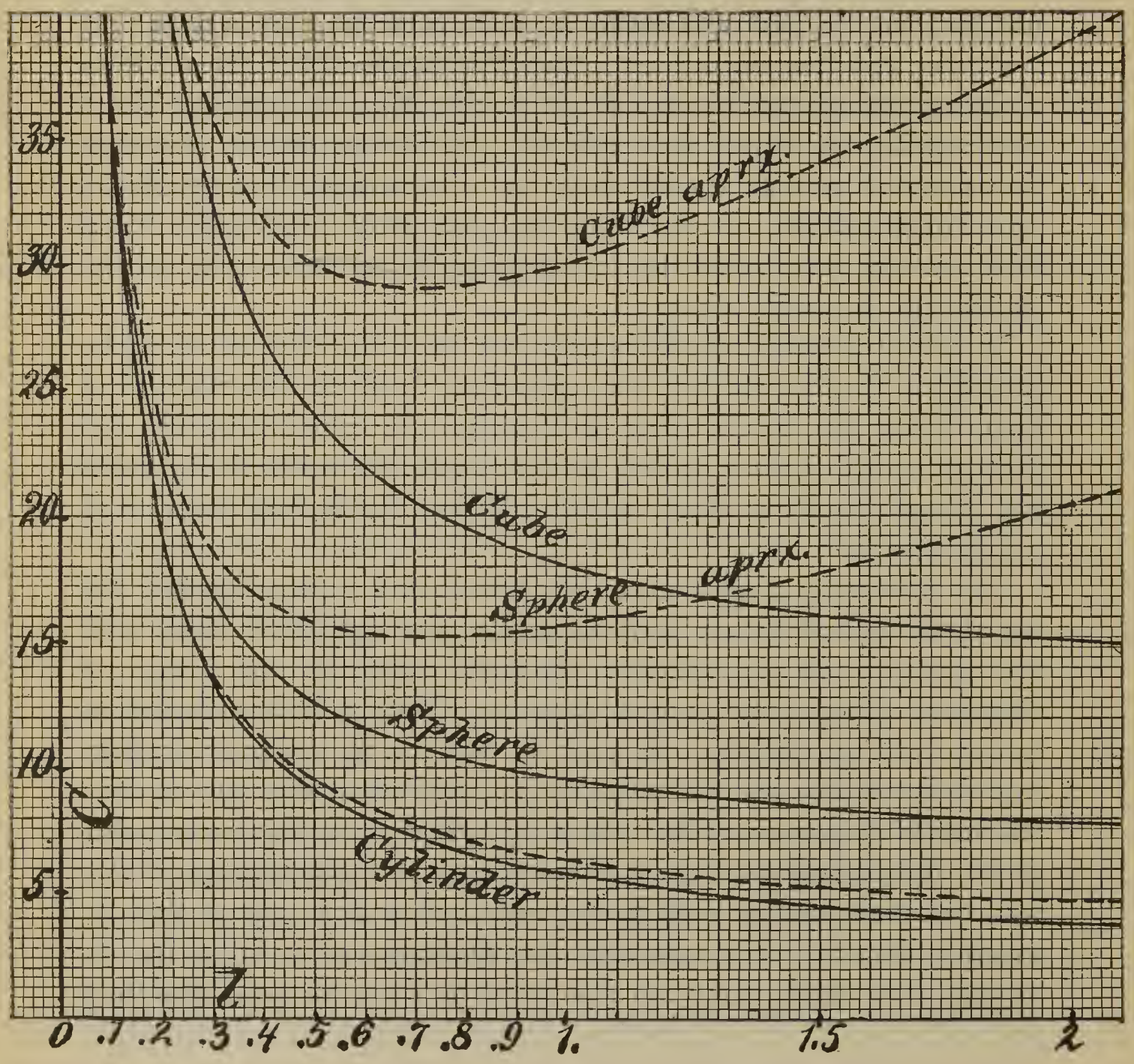


FIG. 4.—HEAT LOSS AS FUNCTION OF THICKNESS OF WALLS.

The curves in Fig. 4 have been so drawn to scale, that they show at a glance how the heat loss diminishes as the thickness of the walls increase. For a diameter of unity, the ordinates are numerically equal to the conductances for a conductivity of unity, that is, they are the numerical values of S/l in formula (1); they

are therefore proportional to the heat losses and need only to be multiplied by the conductivity k, and the temperature drop t, and sometimes by the diameter also, for any given case, to give the actual heat loss.

The distances to the right of the point O, represent the thicknesses l of the surrounding walls in terms of the diameter or of the edge of the cube. Thus it will be seen that for thin walls the heat loss diminishes very rapidly as the thickness increases, but for the thicker ones the decrease of the loss becomes very small; hence a point is reached where it would be of little value to still further increase the thickness.

The reason for this is that the real mean cross section increases very fast for thicker walls, almost as fast as the thickness, and hence the quotient between them, namely, the factor S/l in (1) changes but very little.

Incidentally, it may be remarked here that there are two possible ways of still further decreasing this loss of heat through the walls, which are the diametrical opposites of each other. One is to diminish the temperature drop t, say by heating the outside of the furnace with cheap fuel heat, as for instance by placing the electric furnace inside of a gas or fuel furnace. From formula (1) it is evident that, all others things being the same, the loss is less the smaller the drop of temperature t. The other method is to increase the temperature drop t, say by water cooling the outside. Both methods have been used with apparent success. The explanation of this apparent anomaly is outside of the province of this paper, which deals only with the geometric relations, but apparently is merely a question of whether the product of $k \times t$ in (1) becomes less or greater.

The curves, Fig. 4, also enable one to find directly the value of the thickness l for any given loss, which if calculated would be quite a tedious operation for cylindrical walls, as it involves the equation $ax \log bx = c$. The method of getting this value from the curves will be described below.

Let us now consider the several typical forms more specifically. The spherical type is the theoretically best form as it has the smallest cross section of the insulator (normally to the radius) for a given interior volume. All other forms are worse; the cyl-

indrical comes next and the rectangular, which is perhaps the most common, is the worst.

In the curves, the full line represents the correct results, and the dotted line those obtained from the approximate formula. The latter, it will be seen, differs little from the true curve for small thicknesses, not used in practice, but departs very greatly from it for the more usual range of thicknesses; it even has a minimum point for a thickness equal to about ¾ of the diameter, beyond which the loss would increase again for greater thicknesses, which of course cannot be correct.

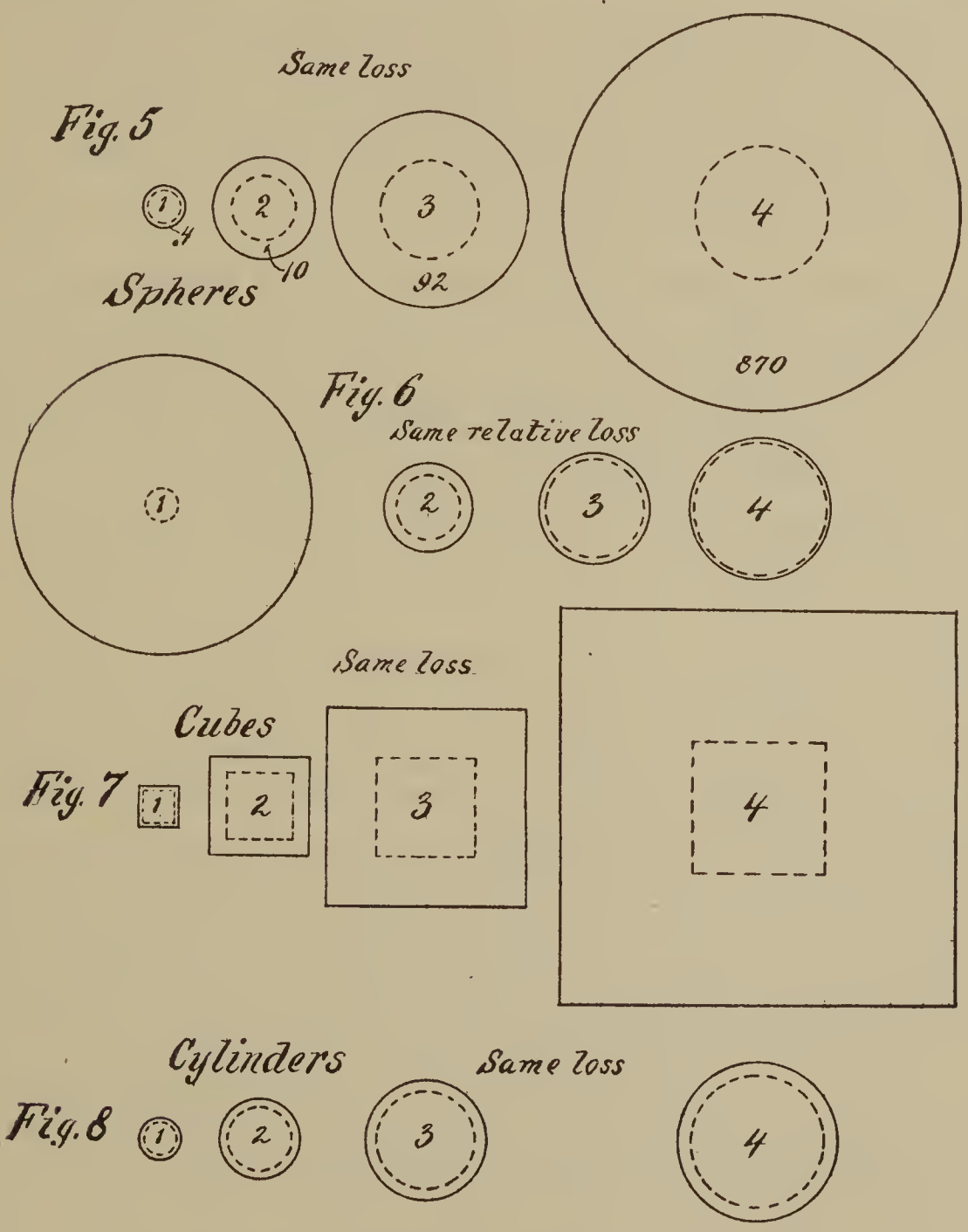
In general the loss calculated from the approximate formulas is the greater of the two, and hence the calculation is on the safe side. This appears to be borne out in practice in which the actual losses were found to be smaller than those calculated with this approximate formula. But the difference seems too great to be overlooked in electrical furnaces, in many of which the predetermined results are a criterion as to whether the actual construction shall be undertaken or not. In a recent case in the writer's practice, the entire function of an electrical furnace was to maintain the material at a given temperature, and not to do any melting or heating; hence the amount of loss through the walls was the only heat to be supplied electrically, and its correct predetermination was therefore of great importance.

The curves show that beyond a thickness equal to a little less than the diameter, there is little to be gained.

In order to show graphically how very important it is to reduce the inside of a furnace to the smallest possible to hold the necessary amount of material, Fig. 5 has been drawn. It shows what the thickness of the walls of a spherical furnace must be when the inside diameter is doubled, trebled, quadrupled, *the actual loss being always the same.* The increase will be seen to be surprisingly great. The volumes of the insulating material are given and will be seen to increase very rapidly, being about 2,500 times as great for the largest one as compared with the smallest one. If these four hollow spheres were made of an electrical conductor, the resistance from the inside to the outside will be the same in all of them.

These diagrams, Fig. 5, are for an equal loss and merely show the great importance of not wasting any room in the inside, be the

furnace a large one or a small one; they do not mean that furnaces should not be made large. The volumes of the interiors of these increase as the cubes of the diameters and hence are as 1 to 8 to 27 to 64. For the same relative loss, considering the increased interior capacity, that is, for the same loss per unit volume, the thicknesses will be shown in Fig. 6. It will be seen



FIGS. 5, 6, 7 AND 8.—COMPARATIVE DRAWINGS, SHOWING SAME ACTUAL LOSS AND SAME RELATIVE LOSS.

that in this case the thickness becomes very great for the smaller ones and very small for the larger ones, becoming nearly constant. The volume of the insulation for the smaller one is even far greater than for the larger ones.

This shows in an equally striking way the very great importance of using as large a furnace as possible, that is, it shows that the loss of heat per cubic foot interior capacity, can be made very much less for a large than for a small one, which means that the heat efficiency can be made much higher for the larger ones. The earth considered as molten mass surrounded by a solid shell, is a good illustration of this case, a relatively thin shell being sufficient to insulate it very effectively. The explanation is that the volume of the inside increases very rapidly with an increase of the diameter, hence the loss per unit volume becomes small. But it must not be forgotten that as Fig. 5 shows, it is very important not to waste any space in it.

It can also be shown from these data that for the same relative thickness, the loss in spherical furnaces increases as the diameter. That is, if the thicknesses in all of the several furnaces is say ¾ of the diameter, the losses will increase as the diameters; but as the cubical contents of the inside increase as the cube of the diameters, the loss per cubic foot of interior space, that is, per unit capacity, will diminish rapidly as the furnaces become larger.

In the comparisons shown graphically in Figs. 5, 6 and 7, it should be remembered that a thick wall must be increased very greatly to make it appreciably better; hence those shown with very thick walls would be nearly as good with a greatly diminished thickness, and should therefore be considered as extreme cases cited to illustrate the laws, and not as giving the best proportions to be used in practice. Instead of using such extremely thick walls, one would naturally sacrifice a slightly greater loss of heat.

Table I gives the numerical values for the spherical type. The first column is the thickness expressed in terms of the diameter d; hence for any particular case these numbers must be multiplied by d to reduce them to inches or centimeters. The next column gives the rigidly correct heat conductances C (for unit conductivity) that is, the factor S/l in the general formula (1), also in terms of the diameter as a unit; hence for any particular case these values must also be multiplied by d. Both l and C are correct numerically for either inches or centimeters, being mere ratios or factors. C' are the corresponding values of C but calculated by means of the usual approximate formulas in which the cross section is taken as the average between the two extremes.

The last column gives the correction factor F, namely, the ratio of the correct to the approximate values; this enables the calculations to be made by means of the simple approximate formula and then multiplied by this factor which will reduce them to the correct values.

To illustrate the use of this table by an example, let the inside diameter of a spherical furnace be 15 inches; let the thickness of the walls be 9 inches; required the head conductance for a conductivity of unity. The relative thickness l/d will be $9/15 = .6$, that is, the thickness is .6 of the diameter. From the table opposite .6 the correct conductance is 11.5; multiplying this by the diameter 15, gives 172.5 in inch units.

If this material is say infusorial earth, having a conductivity in gram calorie, cubic inch units, of $k = .001$, and if the fall of temperature between the inside and the outside is 700°C, then according to formula (1) the heat loss through it will be

$$H = 172.5 \times .001 \times 700 = 121. \text{ gram cal. per sec.}$$

If the relative thickness is not one of those given in the table, or if for any other reason it is desired to calculate the conductance or the heat loss by means of the approximate formula, then multiply the result by the correction factor corresponding to about .6, namely, .75.

Suppose the loss is limited in the above cases to 100 cal. per sec. and it is desired to find the necessary thickness of this material to make it so. The corresponding conductance from (1) must be $100 \div (.001 \times 700) = 143$. Divide this by the diameter 15, giving 9.5. From the table this value of C lies between $l = .9$ and $l = 1.0$, or from the curves it is seen to be about .98, hence the required thickness will be .98 times the diameter 15, or 14.7 inches.

The results for the cubical furnace are given in Table II and in the curves. They are in all respects similar to those of the spherical.

The curves and tables show that the conductances (and therefore the heat losses) will be 1.910 times or about twice as great as those of the spherical form, for the same thicknesses and the same diameter or edge, but it must be remembered that the contents of a cube is nearly double that of its inscribed sphere, hence the difference is not what is seems at first sight.

Comparing a sphere with a cube whose edge is equal to the

TABLE I.—SPHERES

Thickness l ($\times d$)	Conductance C Exact ($\times d$)	Conductance C' Approx. ($\times d$)	Correction Factor F (C/C')
.1	37,699	38.327	.98
.15	27.227	28.169	.97
.2	21.991	23.247	.95
.25	18.850	20.420	.92
.3	16.755	18.630	.90
.35	15.259	17.458	.87
.4	14.137	16.650	.85
.45	13.265	16.091	.82
.5	12.566	15.707	.80
.6	11.519	15.288	.75
.7	10.771	15.169	.71
.8	10.210	15.236	.67
.9	9.7738	15.428	.63
1.0	9.4248	15.707	.60
1.5	8.3776	17.802	.47
2.	7.8540	20.420	.38
3.	7.3304	26.180	.28
4.	7.0686	32.201	.22

TABLE II.—CUBES

Thickness l ($\times d$)	Conductance C Exact ($\times d$)	Conductance C' Approx. ($\times d$)	Correction Factor F (C/C')
.1	72.000	73.200	.98
.15	52.000	53.800	.97
.2	42,000	44.400	.95
.25	36,000	39.000	.92
.3	32.000	35.600	.90
.35	29.143	33.342	.87
.4	27.000	31.800	.85
.45	25.333	30.733	.82
.5	24.000	30.000	.80
.6	22.000	29.200	.75
.7	20.571	28.971	.71
.8	19.500	29.100	.67
.9	18.667	29.466	.63
1.0	18.000	30.000	.60
1.5	16.000	34.000	.47
2.	15.000	39.000	.38
3.	14.000	50.000	.28
4.	13.500	61.500	.22

TABLE III.—CYLINDERS

Thickness l ($\times d$)	Conductance C Exact	Conductance C' Approx.	Correction Factor F (C/C')
.1	34.462	34.557	.99
.15	23.949	24.085	.99
.2	18.673	18.849	.99
.25	15.496	15.708	.99
.3	13.368	13.613	.98
.35	11.841	12.117	.98
.4	10.690	10.995	.97
.45	9.7892	10.122	.97
.5	9.0647	9.425	.96
.6	7.9690	8.377	.95
.7	7.1770	7.629	.94
.8	6.5778	7.068	.93
.9	6.1024	6.632	.92
1.0	5.7192	6.283	.91
1.5	4.5324	5.235	.87
2.	3.9040	4.712	.83
3.	3.2289	4.188	.77
4.	2.8596	3.926	.73

diameter of the sphere, both having the same thickness of walls, it will be found that the loss per unit of volume is the same in both. Hence in this sense the cubical form is no worse than the spherical. A sphere and its circumscribed cube have the same surface per unit volume. But a cubical furnace whose inside volume is equal to that of a given spherical one, will have an edge which is less than the diameter of the sphere; hence for the same relative thickness of walls *(l/d)* the loss will be considerably greater for the cubical furnace; its conducting surfaces will be greater. It follows therefore that for a given interior volume, and the same relative thickness of walls, a spherical furnace is more economical in heat loss, than a cubical one.

For equal losses Fig. 7 shows the rapid rate at which the necessary insulation increases; these figures correspond with those of Fig. 5 for the sphere, and the wall thicknesses are the same.

For the same relative loss, that is, for the same loss per cubical contents, the results correspond with those shown in Fig. 6 for the sphere.

For the same relative thickness the losses are proportional to the length of the edge, like with the spherical form.

The numerical values are given in Table II. Like with the sphere, the numbers in the three colums must be multiplied by the length of the edge, to reduce them to numerical volumes for any particular case.

Lastly, the results for the cylindrical furnace are given in Table III and in the corresponding curves. These are not directly comparable with the others, because they are only for one unit length of a supposedly infinitely long cylinder, and only for the cylindrical part. They must be multiplied by the length of the cylindrical part, and the heat loss at the two ends must be added. Even then there is a slight error at the circular corners, as the results in these tables refer only to a cylinder whose outside and inside axial lengths are the same, as shown in Fig. 2. They are therefore strictly applicable only to long cylinders and do not include the flat ends. They are correct however for comparing different cylinders with each other.

The dotted and full curves show that the approximate formula is much more nearly correct in this case, and it has the same general character. Not only are the actual differences less, but the

percentage differences are less also, as seen from the correction fectors F in Table III. Up to a thickness equal to the diameter, the errors in using the approximate formula are within 10%.

For cylinders it will be seen that the conductances are independent of the diameters, when the relative thicknesses are the same. Hence the values of C and C' in the table are not to be multiplied by d, like in the others. It must be remembered however, that these conductances are for only one unit axial length of the shell and must therefore be multiplied by the length in inches, centimeters or whatever unit is used for the other dimensions.

That the loss is the same for any given relative thickness, l/d, is well shown in Fig. 8, which corresponds with Figs. 5 and 7, and in which the thickness merely increases with the diameter. These however should not be compared directly with those in Figs. 5 and 7, because they are only for one unit of length; the smaller one therefore is a cylinder whose length is equal to its diameter, a so-called square cylinder, because both are unity; while the largest one is four units in diameter, but only one unit long, and represents therefore a mere flat ring.

For the same relative thickness or the same heat loss and the same length, the volume of the insulation will be found to increase as the square of the diameter.

To calculate the total loss in a cylindrical furnace with closed ends, one should first determine the conductances of the cylindrical shell from these tables or curves, and then add that of the ends as found from the tables for the cubical type, which are frustrums like Fig. 3. The sum of these conductances are then used in formula (1) for determining the total heat loss.

The above tables and curves apply not only to the complete enclosures, but also to any parts of furnace walls, that is, to frustrums, like the flat ends of cylinders, for instance. For flat or plane frustrums having different slopes from those forming a cube, the center must first be found by prolonging the slanting sides until they meet; then the quantity d referred to above is twice the perpendicular distance from this center to the inside surface; this must be found in order to determine the relative thickness l/d on which the table and curves are based.

Oblong rectangular furnaces cannot be represented by any one typical form, as they vary in all three dimensions and according

to different laws. Hence no general tables or curves can be given for them. But just as the cubical form follows the same laws as the spherical, so the middle part of a long rectangular furnace (exclusive of the ends) follows the same laws as the cylinder. Hence the conductances of a long rectangular furnace may be calculated by imagining the ends cut off at such a length that when fitted together, they form a cube. The conductance of this part is then calculated as a cube, and that of the remaining central part, as a cylinder. Or the conductances of each of the six sides can be calculated separately as plane frustrums and added together. Until the conductivities k are known with greater precision, great accuracy in calculating these geometric factors of the conductances, is not so necessary.

The above comparisons of the rigid and the approximate formulas, also enable new approximate ones to be determined, which are far more exact over the usual ranges of thicknesses. The rigid ones for the sphere and cube look simple, but they involve a square root determination, which unless done with a slide rule, is tedious; the one for the cylindrical type requires a table of logarithms, and even then it involves quite a little calculating. Approximate formulas which do not involve these objectionable features, and are more accurate than the usual ones, may therefore be of use.

For the correction factors F in Tables I and II, which will be seen to be the same, the following approximate formula is readily deduced for spherical or cubical frustrums or complete enclosures. It gives the conductance C for unit conductivity, that is, it gives the quantity represented by S/l in formula (1).

$$C = \frac{S + s}{2l}\left(1 - .4\,\frac{l}{d}\right) \qquad (9)$$

This is merely the old approximate formula (in which the ordinary average or arithmetic mean is taken for the cross section), multiplied by the simple factor $(1 - .4l/d)$ which is readily determined and will be found to be approximately F of the tables. It is correct within about 1 per cent. between thicknesses of .4 to 1 times the diameter, and within 7 per cent. up to relative thickness of 1.5. Although this may appear at first sight more complicated than the correct one

$$C = \frac{\sqrt{Ss}}{l} \qquad (10)$$

it will be found to involve less calculating as the correction factor is very easily determined. For slide rule calculations the correct formula is too simple to need to be replaced by an approximate one.

Similarly for the cylinder the following may be used

$$C = \frac{S + s}{2l} (1 - .1\ l/d) \qquad (11)$$

This is correct within about 3 per cent. up to thicknesses of about twice the diameter. If the factor .1 is made .085, it becomes very exact for that range. This form is much more convenient than the exact one which necessarily involves a table of logarithms.

ADDENDUM.

Since this paper was written, the writer has noticed the following simplification of the correct formula for the spherical and the cubical types, which avoids the square root calculations.

For complete spheres the outside and inside surfaces S and s may be replaced by their values in terms of the respective diameters, D and d, the larger one D being equal to $d + 2\ l$ and is therefore easily measured or calculated. It can then be shown that the quantity $\sqrt{Ss}$ is equal to $\pi\ Dd$, hence the correct formula becomes simply

$$C = \frac{\pi\ Dd}{l} \qquad (12)$$

which is as simple as could be desired.

This is only for a whole sphere. If any particular frustrum of a sphere is concerned and if it is convenient to express it as a fraction of a sphere, then this same formula may be used, multiplied by this fraction.

Similarly for a cubical furnace the correct formula can be reduced to

$$C = \frac{6\ Dd}{l} \qquad (13)$$

in which D is the outside edge and is equal to $d + 2l$. For any one side it would of course be 1/6 of this or

$$C = \frac{Dd}{l} \qquad (14)$$

which therefore is the correct formula for such frustrums as constitute the sides of a cube.

DISCUSSION.

MR. HENRY D. HIBBARD: Referring to the four diagrams (Fig. 5) representing the four furnaces, I understood Mr. Hering to say that the losses are the same in all; does he mean the same absolute loss or same proportional loss?

MR. HERING: The same absolute loss. The idea of these four diagrams is to show that for a given loss it is of great importance to reduce the size just as much as possible. (Since the paper was read I have added Fig. 6 to show the conditions for the same relative or proportionate loss.)

MR. P. MCN. BENNIE: Considering Mr. Hering's diagrams, from the point of view of one who has to operate furnaces, it is evident that the matter of intelligent heat insulation is very much neglected. We have been running for a long time electric furnaces on the plan here suggested by Mr. Hering, of using an envelope of burning gas as an insulator. There is one type of electric furnace where a gas envelope exists naturally, as in the carborundum furnace. In calculating the efficiency of such furnaces it has often been remarked that there is a very large loss, due to the burning of this valuable gas outside of the furnace. I do not think such is the fact, because the envelope of burning gas is acting as a heat insulator by reducing the difference in temperature between the outside and inside of the furnace.

We have been running for two years or more an electric furnace in which we have intentionally used a burning gas envelope for the purpose of heat insulation, in which the efficiency is very high. The burning gas produces a certain temperature and the heat thus obtained, per dollar expended, is very much cheaper than the heat got by means of electricity, per dollar expended, so

if one reduces the difference in temperature by means of the cheaper method of getting heat there is an increase in efficiency and economy.

MR. C. F. CARRIER, JR.: Mr. Hering's factor k, the coefficient of conductivity, as I understand it, is a variable, and not a constant, that is, as the material cools, this constant decreases. I was not aware that the coefficient of heat conductivity changed with the temperature. When a furnace is water cooled, I cannot see how the loss of heat is reduced. The heat is carried in the cooling water, instead of passing into the air.

MR. HERING: The factor k not only varies, but varies very much, with the temperature, so that it is very necessary, if one expects the calculated results to be at all similar to what one gets in practice, to see that one uses the proper value of k.

MR. JOHN C. PARKER: In regard to the suggestion of Mr. Hering, in reference to the water cooling of the outside of a furnace, I do not quite see how, even with a variable conductivity k, such a beneficent result could follow from the outside cooling, for the reason that in the spherical furnace for example, the inner surface and the outer surface are homologous concentric surfaces of equal temperature. The outside cooling has the effect of pushing the the isotherm which is at the surface with air cooling, back into the mass of the furnace. Now, if we push that isotherm back into the mass of the furnace, immediately we get a greater temperature gradient from the surface to the point to which the initial isotherm has been pushed back. The greater temperature gradient immediately presupposes a greater flow, and that would mean that in order to cool the outer surface we must have a greater flow of heat from the interior of the furnace to the outside.

I would like to ask whether in the insulation of this electric furnace it is not possible to accomplish some economy of heat or economy of material, by doing much the same as is done in the insulation of electrical conductors. The temperature gradient is very steep, indeed, at the middle of the furnace, as shown by the general tendency of these curves. In insulation of electrical conductors for very high pressures, especially with very small wires, it is customary to use at the center of the insulation, an insulating material with an extremely high dielectric coefficient, and outside,

where the stresses are less, the other materials. The question comes up whether it would not be desirable to use graded materials, using materials of low resistance at the center where the flow density is greatest, and at the outside, the less effective heat insulating material.

One other point is that the curves shown here by Mr. Hering—such as that for the sphere—confirm the statement made by him in discussion yesterday, in regard to the resistance between bodies immersed in a conducting fluid. Mr. Hering spoke of two plates immersed in salt water at a certain distance apart. The resistance of the conducting salt water would be represented by such a function as this. If the plates initially are this distance apart (illustrating by pointing out a part of the curve near the "knee") you can move them apart an infinite distance, and yet accomplish a practically negligible increase in the resistance between them, purely and simply because the current density between the plates is reduced to such a very low quantity at larger radii, that increments in the length of the conductor have no influence on the total resistance. That substantiates theoretically very well the statement made by Mr. Hering, which I believe was controverted, and I may say—although this is a little out of order of discussion of the present paper—that practical experiment in the measurement of ground resistances for determining the danger of electrolysis has shown such to be actually the case. I mention this here simply because the curve given nicely expresses the fundamental principle involved in the statement of yesterday.

I might say in this connection—if it is not too distinctly out of the order of discussion—that in connection with a large transmission line we had occasion recently to measure ground resistances. We found with transmission structures twenty feet apart the resistance had reached its practical maximum, that with structures 200 feet, 500 feet, or 1,000 feet apart, there was practically no more resistance between them than in the case of structures only 20 feet distant from one another.

MR. CARL HERING: As to the grading of the insulating materials, it is of course an advantage in all furnaces to do this, that is, to have different materials inside and outside. It would be bad policy for instance to build the whole furnace wall of such low heat resisting materials as chromite or magnesite bricks; such

bricks must be used for as small a part of the wall as possible. The materials must be selected in each case for the specific furnace.

Concerning the heat gradient, the whole thing is a matter of calculation. If you find that by cooling the outside of the furnace the product *kt* becomes less, that is the loss becomes less, then that is the proper thing to do. But if you find that by heating the outside this product *kt* becomes less, and the loss therefore becomes less, then that is the proper thing to do; in other words, it is a question of simple engineering calculation as to which is the best to do under the specific conditions given, provided one has the data to make this calculation.

Prof. J. W. Richards: There is needed for the use of metallurgists and electro-metallurgists a large series of determinations of just these quantities of heat conductivity of different materials at different temperatures. As far as I know, there is very scanty data on the subject. There are probably not more than three or four determinations of the heat conductivity of different temperatures, which we can use, and I should very much like myself to have six months at my disposal, so that I could go into the laboratory and work on these things, and work out their application to the different furnaces; but we need first of all the purely physical experimental data curves, determined in the laboratory, and then we need some clear exposition of the principles, such as we have had here, and much more could be added to it, to show how the principles can be practically applied to the metallurgical art.

I think there is a maximum wall thickness with which you will have the maximum protection. I came across that recently in studying the heat radiation from a certain sort of furnace. It was shown by experiment that after a certain thickness was reached there was no more gain in putting on protecting material outside, as the increased effect from the additional material was practically negligible.

Mr. F. A. Lidbury: I think there is one interesting point clearly brought out by the diagrams Mr. Hering has drawn, and that is the danger of making simplifying assumptions in calculating the magnitude of heat losses in the electric furnace. If one refers to the diagram Fig. 4, in the case of the sphere and cylinder,

one observes at once the extreme disproportion between the size of the walls in these two cases, and yet it is barely possible to conceive of a case which is represented strictly by any of these diagrams. The actual cases will be usually found to lie somewhere in between, and the extreme disproportion shown by these diagrams, to my mind, indicates the caution with which such simplifying assumptions should be applied in practice.

MR. CARL HERING: I should call attention to the fact that for the cylinder these diagrams represent only the loss per unit length. It is very important to remember this, because if you were to design a cylindrical furnace of a certain diameter and length, and calculate the heat losses from the total outside surface, the ends included, you would probably find that the walls would have to be even thicker than in the sphere, because the sphere is the theoretically best case. It must not be forgotten that the curves given here for the sphere and the cylinder cannot be compared directly, but that the comparison must be made per unit of length of the cylinder, which is the only way such curves can be given for the general cases.

MR. W. S. LANDIS: As to the matter of data on this subject, I might mention a reference book, which may not be well known, but which contains a large amount of data on the subject of heat conduction of different materials, and that is Peclet's old book, "La Chaleur," which I think it would be well for many of the electrical and other engineers to read. He has worked out this problem to a large extent, and has furnished a very great amount of data on the subject of the conduction of heat. Fourier also worked over this same matter.

A paper read at the Fourteenth General Meeting of the American Electrochemical Society, at New York City, October 31, 1908; President E. G. Acheson in the Chair.

THE LASH STEEL PROCESS AND THE ELECTRIC FURNACE.

By F. A. J. FitzGerald.

In this paper it is not intended to do more than give a description of the general principles of the Lash process, and describe briefly certain experiments on its application to the electric furnace. There are certain misconceptions as to what the Lash process is, and also it is not obvious at first sight why it might be of interest to the electrometallurgist. As regards its use in the open hearth, little need be said here beyond mentioning that the experiments, made on a commercial scale at several steel plants, have been successful in demonstrating its usefulness. As might be expected, a process of this kind inevitably gives an opportunity to the person who is convinced that he did the same thing twenty or thirty years ago, but discussions on that subject are of more interest to the archeologist or mythologist than to the electro-metallurgist, and in any case are not usually profitable.

The broad principle of the Lash process consists in making an intimate mixture of ore, cast iron and carbon, and submitting this mixture to heat, when, if the proportions of the constituents of the mixture have been properly adjusted, the desired grade of steel is produced. The essential features in the process seem to be the fine state of division of the constituents of the mixture, their intimate association and the use of iron containing metalloids in considerable quantities.

It is unnecessary to comment on the advantage of having the various reagents in a fine state of division and intimately mixed, for the value of such a condition may be readily appreciated; but the use of iron, containing metalloids, is worthy of more extended notice. According to Lash:[1]

[1] U. S. Patent 860,922, December 28, 1906.

"The action of the pig or cast iron in the finely divided mixture is two-fold in character: First and primarily the pig iron contains a high percentage of metalloids and, generally, manganese, which elements upon the fusion of the iron, are presented to the oxid in the most intimate and effective condition possible, and become active and ready reducing agents. Secondly, the fused pig iron seems to act simultaneously as a solvent for some of the free carbon, and as an enveloping coat for the individual particles of oxid, thus bringing such dissolved carbon into intimate association with the oxid to supplement the effect of the metalloids already contained in the pig iron, in their reducing action."

Thus, according to this hypothesis as to the working of the process, the finely divided iron acts as a carrier of the carbon, and the reactions might be represented by an equation as follows:

$$3\ Fe_3C + Fe_2O_3 + 3\ C = 3\ Fe_3C + 2\ Fe + 3\ CO$$

It follows then that during the reaction all the iron present in the metallic state contains carbon in excess of that required in the finished product, till towards the end, when the free carbon is completely exhausted, and there is simply a mixture of iron high in carbon and unreduced oxide. Finally this mixture reacts, and the desired quality of steel is obtained. While the reactions which actually take place are no doubt far more complicated than this hypothesis of the working of the process, yet the experiments which have been made seem to demonstrate the usefulness of the supposition.

In practice there are many details which have not been mentioned, as, for example, the addition of fluxes such as lime and fluorspar to the mixture; the use of sawdust which is carbonized in the furnace and leaves the mass in a porous state so as to permit of the ready escape of the gases formed; the use of coal tar or any other suitable material to prevent the separation of the constituents of the mixture from each other because of their widely differing specific weights or to act as a binding material in briquetting. These details, however, need not be considered at present.

In the application of the Lash procees to the open hearth furnace the saving effected is due to the lower cost of a unit of iron in the form of ore, than in that of pig or some forms of scrap.

From this it follows that, other thing equal, the less pig iron and the more ore that can be used the cheaper will be the steel produced. As to the arguments which may be advanced, that the process cannot compete with an open hearth furnace fed with molten pig iron, or with other modifications of the open hearth furnace, they are foreign to the present considerations. The expert steel maker may also discuss the correctness of the observation that steel obtained directly from a charge containing a much larger excess of ore than is possible in the ordinary open hearth method possesses superior qualities to that manufactured in the usual way. The contention may be difficult to support *a priori;* but some empiricists are nevertheless convinced of its truth, and even when they admit the possibility of making good steel from any kind of raw material make inconvenient inquiries as to what it costs.

That it is possible to produce steel directly from iron ore in the electric furnace is well known. This has been done by various electro-metallurgists, but the best published account of that work is by Major Stassano. According to his most recent publications[2] the best results obtained in the production of steel, where the charge consisted essentially of ore and carbon, showed an energy consumption of 4 kilowatt hours per kilogram, or 0.62 horse-power year per gross ton. The cost of lining the furnace, as calculated from a long series of experiments, amounted to $1.93 per ton (metric?) of output.

The experiments made with the electric furnace in the production of pig iron at Sault Ste. Marie, under the auspices of the Canadian Government, have shown the possibility of producing a ton of metal from a mixture of ore, carbon and fluxes, with an expenditure of less than 0.28 horse-power year. Now it seems reasonable to suppose that if by using the Lash process in the electric furnace an energy consumption comparable with that of the electric pig iron furnace could be obtained the field of usefulness of the electric furnace in the metallurgy of iron and steel would be greatly extended. One of the most serious difficulties experienced in all processes where the reduction of iron from the ore with the object of producing steel directly is aimed at, is the serious loss of iron which occurs, so that the yield of metal is

[2] Electrochemical and Metallurgical Industry, Vol. VI, No. 8, p. 315, *et seq.*

too small. So far, however, as the Lash process is concerned there was little reason to fear a difficulty of this kind, for in the preliminary experiments made at our laboratories on a small scale it was found that 98 per cent. of the iron contained in the mixture could be easily obtained. This may be readily seen when it is pointed out that in these experiments the metal and slag were carefully preserved and weighed, and that it was found that the total loss of metal in the slag only amounted to 0.3 or 0.6 per cent. of the iron contained in the mixture put in the furnace. So far, therefore, as the electric furnace was concerned, the chief point to be determined was the amount of energy required to obtain one ton of steel.

In order to investigate this matter there was a choice of various furnaces, for it was decided that to get results of any value it was necessary to work on a scale which would produce steel in commercial quantities. The possible furnaces seemed to be those of Girod, Héroult, Keller, Röchling-Rodenhauser and Stassano. For various reasons it was decided that the Niagara Falls experiments could be carried out most conveniently and cheaply in a Héroult furnace. Accordingly a 3-ton furnace was constructed under the direction of Mr. Robert Turnbull and an experimental plant installed on the land of the Niagara Falls Power Co., from whom the necessary current was obtained, and who gave us much valuable assistance in preparing for the work.

With this plant there were manufactured about 50 tons of steel having various analyses, of which a few are given in the following table:

Heat No.	9	11	23	27
Carbon	0.10	0.08	0.92	0.22
Manganese	0.75	0.09	0.41	0.94
Phosphorous	0.015	0.015	0.038	0.033
Sulphur	0.070	0.007	0.026	0.056
Silicon	0.02	0.09	0.13	0.03

The following tests were made from bars 1 inch square, hammered from 6 x 6-inch bottom cast ingots. The elongation was determined in a length of 2 inches.

Heat No.	Tensile Strength.	Elastic Limit.	Elongation per cent.	Reduction per cent.
9	57000	34000	33	58
27	77000	45000	28	56

The steel of heat N° 27 was treated with 0.1 per cent. of titanium.

In all twenty-six runs were made, the methods of working the furnace being varied so as to investigate the working of the Lash process under as widely differing conditions as possible. Naturally such a method of experimenting was not conducive to the highest efficiency of working, but nevertheless we were able to obtain complete reduction of the charge with an expenditure of 0.27 horse-power year per ton of steel, and turned out a finished product with an energy consumption of 0.33 horse-power year per gross ton of steel cast. In all the experiments there was an excessive expenditure of energy in the process of finishing the steel, adjusting the carbon, removing phosphorus, etc. Making due allowances for the difficulties under which the experiments were carried out, such as inadequate apparatus, inexperienced workmen, variation of the method in every experiment, it is believed that under suitable conditions a production of four tons of steel per horse-power year can be reached.

The lantern slide shows the furnace in process of pouring the steel into the ladle. We are indebted to Mr. J. R. Wilson, who did most of the construction work on the plant, for this excellent photograph of a very difficult subject.

A serious problem met with in the working of the process in the Héroult furnace was the wear of the carbon electrodes, which in many runs was excessive. This was found to be due to various causes, but more especially to the inadequate apparatus for adjusting the electrodes and to certain faults in the method of working the furnace. Experiments made with a view of correcting the latter cause showed such an astonishing decrease in the electrode consumption that it is reasonable to believe that while the best results obtained in the experiments showed a consumption of 69 pounds per ton of steel cast, this may be brought below 50 pounds per ton.

FIG. 1.—HEROULT FURNACE TAPPED.

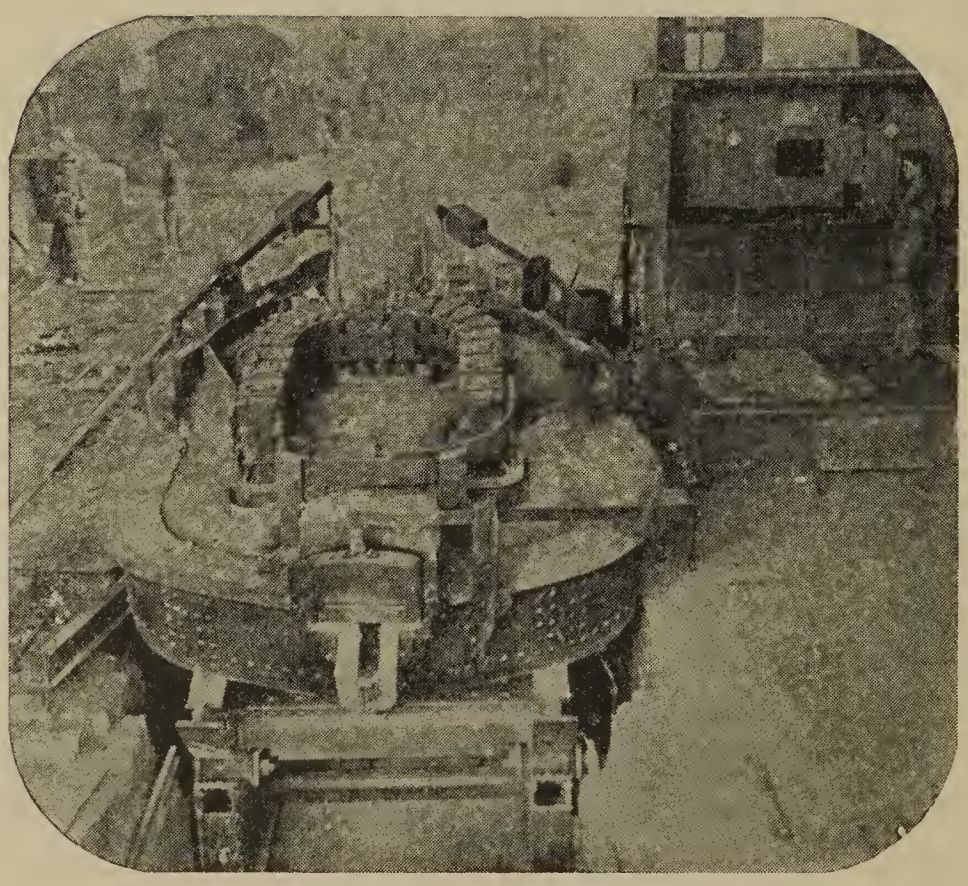


FIG. 2.—THREE-PHASE ROECHLING-RODENHAUSER FURNACE.

Another very important consideration in the process is the wear of the lining; unfortunately there has not been a sufficient number of runs made to obtain an accurate estimation as to what this item will amount to.

In carrying out the Lash process in the electric steel furnaces of Keller and Girod the conditions would be very similar to those met with in the Héroult furnace; but in the Stassano furnace the electrode problem would probably be much simpler since these are held some distance above the charge. Whether the consumption of energy would be as low as in the Héroult furnace is another question; but it seems certain that the cost of producing steel would be less were the Lash process used than when working with the simple direct method.

While the electrode problem can, no doubt, be successfully solved, its existence emphasizes the interest of a furnace which can be worked without these troublesome and expensive appendages. The Colby-Kjellin induction furnace would obviously be unsuitable for use with this process, because it does not permit the handling of slags in a convenient and economical manner, but the modification of this apparatus, known as the Röchling-Rodenhauser furnace, might be expected to give interesting results The next lantern slide shows a three-phase furnace of this kind, the three channels forming the secondaries of the induction furnace being connected to a central bath which is so constructed that the slag formed during the reduction of the ore can be handled. Moreover, this central bath would also permit of the easy introduction of the charge, which would probably be in the form of briquettes.

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DISCUSSION.

W. S. Landis: On your experimental runs, in which you obtained a ton of metal with a power consumption of 0.270 h. p. years per ton of metal, did you reduce the iron content of the slag to 2 per cent.?

MR. FITZGERALD: In that particular case, I do not remember exactly what the analysis of the slag was. We made several analyses of slags, and at first found that we were losing a good deal of metal, in some cases the metallic content of the slag being as high as 15 per cent. However, in the later runs, the loss through the slag amounted to only about 6 per cent. metallic iron, and as the low power consumption was obtained in the later runs, it is probable that in the runs I referred to, where the power consumption was low, the loss of iron in slag was low also.

HENRY D. HIBBARD: This whole question of the electric fusion of iron is intensely interesting, and the paper just read is a very valuable contribution to the subject. There is one thing which may be new to some of the members, and that is the great advantage of the electric furnace in the production of heat without oxidation. When you make steel in the open hearth furnace, you produce all the effects by oxygen, the heat by its union with the fuel, the elimination of the carbon and silicon, and in fact it is the great servant you use in producing the results, but oxygen, while an excellent servant, always lies in wait to give you all kinds of trouble, if you do not handle it right at the latter end. The oxygen must be fairly eliminated from the metal or the result is poor. In the electric furnace you can produce heat without oxygen, and from a steel maker's point of view the process of making steel by such furnace is simplified more than you can realize unless you have been through it yourself.

To get rid of oxygen, both as an agent and a disease to be cured at the latter end of the process, is a very great point, which, as far as I know, pertains to all electric furnaces. The induction furnace meets this point especially well. Metallurgically the production of steel in the induction furnace is very simple, and the only difficulties I foresee are those of capital required and the investment.

J. W. RICHARDS: There are two ways of making steel, one is simply to melt the ingredients together, and the other is to oxidize the pig iron so as to eliminate the impurities and get down to steel. These two processes require essentially different kinds of apparatus, for the reason that in the melting process it

is simply a case of melting; in the oxidation process, it is largely a case of having surface to oxidize the bath by means of the iron ore which you add, so that the requirement is that there should be considerable surface so that you can work to oxidize out the silicon, etc.

These two requirements are going to give rise to two different classes of furnace, one best adapted for one purpose and the other best adapted to the other purpose.

As to the question of losing iron in slag, that is not such a very serious one, because in ordinary open hearth processes there is considerable loss in slag. I would not want the electrochemists to think that a process to be commercially successful must reduce the iron in the slag to a low point. There are open hearth furnaces running, making steel successfully, for instance the Monell process, where they lose something like 15 or 20 per cent. of iron in the slag, but yet are commercially successful, so that is not a *sine qua non* to get the slag low in iron, so far as saving the iron is concerned.

The third point that I wish to make is that the electric furnace is being developed very largely by electricians and electrochemists, who in many cases are not experienced steel men, and the ideal combination is to get the experienced steel man with a first-class electrochemist to work on the proposition. I notice that where a firm which is experienced in making steel secures the assistance of an electrochemist or an electro-metallurgist to do the electrical work for them, that the combination succeeds very nicely. There is a great deal to learn about the different steels; it takes a lifetime to know all the fine points of them, and the electro-metallurgist cannot jump in and make steel right off, without running into a large number of pitfalls which the practical steel man will be able to sidetrack for him. The practical steel man has a good deal to learn about the electric furnace, and the electrochemist has a great deal to learn about steel, and I would strongly urge the combination of the two.

MR. LANDIS: In regards to Prof. Richards' remarks about the loss of iron in slag, as I understand the Lash process, it is intended particularly for the reduction of iron ores and the final production of steel directly from them in the same operation. It seems to me, therefore, that the loss of iron in the slag is

rather a case of the old blast furnace, in which we had to clean the slag as much as possible, and then refine the material after it has been reduced. It seems to me, therefore, the loss in slag is quite a serious operation in that case, if I understand the process aright.

W. H. WALKER: Will Mr. FitzGerald give us in a few words what the Lash process is?

MR. FITZGERALD: The Lash process consists in making an intimate mixture of cast iron, oxide of iron and carbon. The cast iron is in the form of borings or shotted pig iron, and is supposed to act as a carrier for the carbon, and also the metalloids contained in it, undoubtedly act as reducing agents. The process seems to be extremely simple, and several people have already said that they have worked it before, but it is rather strange, if that has been done, why the work has almost always stopped after apparently a very small experiment, whereas the Lash process has been carried out on a very large scale, many hundreds of tons of steel having been made in the open hearth process, showing economies over the regular practice.

MR. RICHARDS: With that explanation, I would call attention to the similarity of the Monell process, which I mentioned, which takes melted cast iron and uses iron ore to oxidize out the carbon and silicon, and in that case the slag is high in iron and runs at 35 per cent. of ferrous oxide, but that loss is tolerated, because of the other advantages of the process in getting rid of phosphorous, and in other respects, but essentially it is a refining process, such as that which has been explained.

MR. FITZGERALD: I think in the Lash process a much larger percentage of ore can be used, and in the open hearth furnace there is no difficulty in reaching a yield of 94 per cent., which means a loss of 6 per cent. in the metallic contents.

MR. RICHARDS: What percentage of iron ore is used with the cast iron?

MR. FITZGERALD: I have not the figures in the open hearth process. One method of working has been to have a bath in the furnace, and then add the Lash mixture. In the electric furnace, in the experiments we made, the mixture contained about

60 per cent. of ore, about 23 per cent. of cast iron, and the remaining constituents were fluxes and coke.

MR. RICHARDS: That is a larger proportion of ore and reducing agent than is used in the Monell process. The Monell process uses melted pig iron and ore.

MR. HENRY D. HIBBARD: Speaking about the Lash process, I was struck with the apparent similarity of principle between that and the old Uchatius process, that was developed and worked to some extent in Austria about fifty years ago, only in that process the melting was done in crucibles, and in the present case it is done in large furnaces. If I recall correctly, the ingredients used in the Uchatius process were finely divided iron and iron ore, and the melting was done in plumbago, that is, carbon crucibles

A paper read at the Fourteenth General Meeting of the American Electrochemical Society, at New York City, October 30, 1908; President E. G. Acheson in the Chair.

CALCULATION OF THE LATENT HEAT OF VAPORIZATION OF ZINC.

By Woolsey McA. Johnson.

I have spent the best part of five years and a half studying the condensation of zinc vapor, with a view to meeting all sides of the problem—scientific, commercial and practical. For this reason, in November, 1903, I calculated the latent heat of vaporization by use of thermodynamics as follows:

(1) $A - U = T \frac{dA}{dT}$ by second law of thermodynamics

(2) $A = p\,(V - V')$ work $=$ { pressure × change in volume

(3) $\frac{dA}{dT} = \frac{(d\,p\,V - Vi)}{dT}$ { differentiating (2) with respect to T

(4) $U = p\,(V - V') - l$

(Total energy supplied) = (work done in distillation) − (energy lost in distillation)

l = latent heat of vaporization.

(5) $l = T\frac{dp}{dT}(V - V')$ by combining (1), (2), (3) and (4) in terms

In equation (5)

l = latent heat of vaporization in litre-atmospheres

T = the absolute temperature of boiling at 760 mm. pressure

$\frac{dp}{dT}$ = increase of pressure needed to keep zinc liquid for increase of temperature of one degree centigrade at 920° C

$T = 920 + 273 = 1193°$ absolute

$\frac{dp}{dT}$ was determined by Barus at 920° C. to be nearly $\frac{1}{1.5 \times 76}$.

Bulletin U. S. Geol. Survey No. 54—Mar. 10, 1889.—Determinations made by me when working for Lungwitz Reduction Co., of "pressure-temperature" curve of liquid zinc up to 75 lbs. absolute per square inch.

Throwing out such of the determinations as were clearly erroneous, we have a probable value at 920° C. (which I have found to be the boiling point of zinc at 760 mms. pressure of mercury), of 0.152 lbs. per square inch increase of pressure for 1° C. increase of temperature.

Using my determinations for the calculation we have

$$0.152 \text{ per square inch} = \frac{0.152}{0.193}$$

$$\text{or } \frac{dp}{dT} = 0.784 \text{ in } 0° \text{ C. } 8 \text{ cms. of Hg.}$$

$$= \frac{0.784}{76} = 0.01035 \text{ in } 0° \text{ C. and atmospheres of } 76 \text{ cms. Hg.}$$

$V' = \frac{0.001}{6.5}$ litres specific volume of liquid Zn at 920° C. (so small that it can be neglected.)

V = specific volume of zinc vapor at 920° C.

$$\text{V. D. of zinc } (H = 1) = \frac{65.4}{2} = 32.2$$

$$\text{V. D. of zinc } (\text{Air} = 1) = \frac{32.2}{14.4}$$

$$= 2.2$$

Specific volume of air at 920° C. $= \frac{1193}{273} \times \frac{1.00}{0.0896 \times 14.4}$

Specific volume of air at 920° C. $= 3.42$ litres

Specific volume of zinc vapor at 920° C. $= \frac{3.42}{2.27} = 1.51$

Putting in equation (5) all values we have

$$1 = \frac{1193 \times 0.01035 \times 1.51}{}$$

$= 18.6$ litre-atmospheres

$= 18.6 \times 24.25$ Cal. for one litre-atmos. is equal to 24.25 cal.

latent heat of vaporization $= 451.$ calories for one gram of zinc.

Making calculation on Barus' figures, which I believe not as accurate as mine, which represent 300 or 400 measurements, we have for L. H. V. 388.

Prof. J. W. Richards page 447 *et seq.* Trans. Am. Elec. Chem. Soc. Vol. XIII calculates L. H. V. of zinc by Trouton's law to be 426. It can therefore be assumed that the mean of 426, 451, and 388 or 422 cal. is a figure close enough for practical purposes to the actual figure.

I made a similar calculation for L. H. V. of mercury using figures in Landolt & Bornstein and obtained 61.8 cal. Landolt & Bornstein Tabellen, page 347, give 62.0 for the figures as determined by Person in 1846.

For a fuller exposition of the equations, the reader is referred to Nernst, *Theoretischen Chemie,* zweite Auflage, p. 62, 63, 64.

In order to condense zinc practically on a large scale, it is necessary to study the physical chemistry of the problem and also to have due regard for the engineering and commercial side. The latter is, of course the acid with which to test your theories.

A paper read by C. A. Graves at the Fourteenth General Meeting of the American Electrochemical Society, at New York City, October 31, 1908; President E. G. Acheson in the Chair.

POWER FOR ELECTROCHEMICAL INDUSTRIES.

By John Meyer.

The tendency of the times is in the direction of specializing. Special effort in any one direction results in approaching closely to perfection in any development. This is no less true in the electrochemical and the central station industry than in other lines of business. If it were possible to eliminate from any process all of the factors incidental to the production such as, for instance, the supply of current, then it is fair to assume that the maximum effort could be applied to the actual process. All things being equal, there is then every incentive for co-operation between the electrochemist and the central station even to the extent of the location of the electrochemical plant with reference to the central station and hours of use of the current.

With the rapidly increasing demand in this country for electrochemical products, as is evidenced by the importations, it would appear important, if not absolutely essential, to develop this industry here and to encourage relations between the central station and such industries at this time.

This is particularly true in the matter of development wherein the one hinderance is the large immediate investment in order that an experiment may be conducted. Considering then that the experimental load can be so adjusted as to fill valleys which exist consequent to the conditions of the central station load, there is then the possibility of obtaining a low rate depending upon local conditions, which, however, would be mutually satisfactory to both parties concerned, and which would help to develop the industry.

The majority of the industries started under circumstances where the service supply of the central station could have been used to advantage.

The maximum possible output of any plant cannot be maintained continuously. The actual output is a certain percentage of the maximum and may be considered as the average. The ratio between the maximum and the average may be expressed in terms of load factor as illustrated in the following example: If a plant having a maximum capacity of 100 units per day, and the average represents 60 units, then the load factor may be represented as 60 per cent., indicating that the average output was 60 per cent. of the maximum possible. If the current required to perform this operation is in direct proportion to the output of the product, then in the example stated above it is fair to say that, although the maximum may be required at intervals, the average load will only represent 60 per cent. of this maximum. Applying this principle to the rate of $30 per hourse-power per annum of the capacity, because of the average load being but 60 per cent. of this capacity, the cost of current becomes actually $50 per horse-power per annum.

This question of load factor is one of importance and has not been fully appreciated by the electrochemical industries. Quoting from figures given recently in the French Journal, which is the authority upon electrochemical industries abroad, it is learned that the actual load factor of the plants on the other side varies from 50 per cent. to 60 per cent., based upon 24-hour 365-day year. In other words, the cost per horse-power is not the actual cost divided by 365 times 24, but it is a figure which is obtained by multiplying the equivalent 8,760 hours by the load factor in percentage, and this resulting figure divided into the annual cost per horse-power, will produce the actual cost per horse-power hour.

The matter of location of an electrochemical plant is materially affected by facilities for obtaining the raw material or of the disposal of the finished product. For instance, the ideal location the the plant, other things considered equal, would be such that the raw material may be obtained immediately adjacent to the plant and the finished by-products disposed of immediately adjacent to the plant. In a recent investigation of contemplated electric furnace plant, the central station offered reasonably low rates for an experimental plant of small capacity, holding out as an inducement, which has been admitted to be equitable, a reason-

ably lower rate in the event of the furnaces proving satisfactory, and the furnace capacity increased to such an extent as to eliminate all of the oil furnaces now in use. There is every evidence that if this particular installation proves satisfactory, that a great many similar furnaces will be installed in various industries.

In another instance a small arc furnace having a capacity of 20 k.w. has been installed for the purpose of demonstrating the production of nitrogen compound with the electric furnace. The current for the operation of this furnace is purchased from central station. Upon completion of this demonstration, a series of furnaces of considerably larger capacity will be installed.

From what has been said in this and in preceding papers it is evident that the central station is working up to the possibilities in this line, and it may almost be truly stated that they are keeping pace with the demands of the electrochemist.

From the very nature of the carborundum process, requiring as it does, 19 hours' operation of the electric furnaces, after which the furnace is broken down and rebuilt, it may be said to be an ideal process for central station operation. Such a process could well be located where the by-product could be utilized in the manufacturing industries, and where the sale of this by-product may offset to a very large extent, if not in total the cost of electric current.

The various phases of this question have been discussed in various papers published within the last few years, and there is very little to be added at the present time.

A paper read at the Fourteenth General Meeting of the American Electrochemical Society, at New York City, October 31, 1908, President E. G. Acheson in the Chair.

UTILIZATION OF POWER STATIONS FOR ELECTROCHEMICAL AND ELECTROTHERMAL PROCESSES DURING PERIODS OF LOW LOAD.

By E. A. Sperry.

With reference to the utilization of idle loads of central stations for electrochemical and metallurgical work, I have briefly to add to the remarks I have made on former occasions on the same subject some items which I hope will be of interest, which have crossed the horizon since our last meeting. It is gratifying to note the increased interest in this subject on the part of central station managers. The meeting arranged for by the Brooklyn Edison Company as between representative central station managers and some representatives of the electrochemical and metallurgical industries, has already been noticed in the "Electrochemical & Metallurgical Industry," in the current number. I had the pleasure of being present at this meeting and it seemed to me that I noted an undertone of disappointment at the low prices which some of the metallurgical and electrochemical industries are receiving, as, for instance, the price on the Canadian side at Niagara Falls; power in blocks of some considerable size has been placed at quite considerably less than $12.00 the horse-power year. Yet it may be said that the station managers—especially in the larger centres—may possess an advantage in the line of transportation, freights and accessibility to the market over plants located in more remote parts; although, on the other hand, it must be remembered that any tendency to intermittent service will encroach considerably upon these advantages. I wish to emphasize a point which has been heretofore mentioned in this connection, and that is, that it seems to me that much good will come from a continued agitation of this subject; our electrochemists and electrometallurgists must be taught to think, insofar

as possible, in intermittent processes—processes that will not be greatly damaged by cessation during certain hours, for certain fractional parts of a year. Undoubtedly such processes can be developed and products produced economically along these lines. The importance of this should be emphasized, and we should bear in mind the importance of this matter to the station managers, for it is doubtless a fact that to secure the very greatest advantage to all parties the station manager should practically have control of the operation of the electrical or metallurgical plant. It is a fact that in this city upon the sudden approach of a severe storm, when the city is overcast with dark clouds, the sudden demand for current may extend to a great many 100 per cent. of normal load at the particular hour. Upon such occasions it might become necessary for the central station to gather in all the resources possible in the line of energy output for its lighting service.

Again, since our last meeting, the details of an extremely large contract for power have been made public in Chicago. The prices here ruling give us some clue as to what may be expected from central station service. These prices, as we know, are not far from $30.00 per horse-power year. Another point which should be emphasized in this connection in the line of caution is the selection of the process. It should be one for the product of which there is almost unlimited demand, and at the same time one where the power cost constitutes a large or prime factor. In this line many processes will occur to you.

A plant originally laid out by Mr. Scott, past president of the American Institute of Electrical Engineers, has come to my attention, in which there have been successfully combined an electrochemical process and the ordinary central station load. This is a plant for the production of caustic soda and bleach, in southern Maine, and has now been in successful operation for about three years. Here the surplus current is used upon the electrolytic cells, and at times of high load the current of these cells is cut down and energy utilized for light and power purposes, but it is noted in this instance that it is seldom that the current on the cells is cut down to more than half load. This would not be considered as giving anywhere near as much flexibility as is desired. If the current in this instance

could be cut down to one-tenth or even better, to one-twentieth, or 5 per cent. of the original load, for purposes of preventing "reversal," it would be far more desirable from the standpoint of the central station. Other instances of this kind may have come to the attention of our members, which I hope they will mention in the course of the discussion.

I see that we have with us this morning a number of the prominent central station men, whom I am sure we welcome in our midst, and hope they will contribute liberally to the discussion of this very important matter.

DISCUSSION.

PRESIDENT ACHESON: This seems to be a subject that ought to be of great importance to electrochemists. There is one error, I think, in the paper in regard to the duration of the run of carborundum furnaces. It is thirty-six hours, so that it would overlap all the high points and valleys, and is not available for central station work, but there should be many industries that could be fitted in; but not so largely in the case of a furnace, because, after we once get the furnace fairly heated up, we cannot very well let it go out, because it represents a large amount of energy, the initial heat in warming up your work, however, for the electric current at the times when the load on the station is low.

MR. EDWARD R. TAYLOR: I spoke some time since concerning a paper by the same author, in which I made some criticisms. I am of the same opinion still, unless proper arrangements can be made, and I am glad to see that overtures are being made by the producers of power, by which they are willing to take the initiative in interesting electrochemists and others in such work. If that can be done, there are many places in which that work can be done. My own furnace is capable of intermittent work in that respect, but, of course, not to as good advantage as though running continuously for twenty-four hours. It would not be advisable to shut the current entirely off during the peak load. For example, if you run twelve hours and then stop twelve

hours, you would barely get started again. On the other hand, on the peak load the furnace could be kept hot, and just below the point of production, and then when the valley load came an increase of power could be put on and the production proceed for twelve hours. I do not mean to say that would be as advantageous as the continuous twenty-four hours' production, but, on the other hand, if the power supplied were sufficiently cheap to justify it, the furnace I am running is an illustration of the possibility of utilizing such product. In that particular case it would be impossible to use it in cutting off the current entirely for twelve hours, but we could probably cut off for the two or three hours of the peak load. Such use of electricity in these stations might be very desirable for certain experimental work, which work might be arranged to be done during the valley loads on the central station, work which could be done at that time, but, of course, that would be a minor item.

MR. E. A. SPERRY: Mr. Taylor is a man who has had a great deal of experience, and I think this a good opportunity to get this thing into concrete form. Suppose your peak was only five hours long, and you had current supplied nineteen out of the twenty-four hours, then in what ratio could you cut down; could you cut down four-fifth or nine-tenths; how much current would you want to bridge over the peak; how low could you get that down in proportion to normal load?

MR. TAYLOR: I think we could take one-quarter the maximum.

MR. SPERRY: And give up three-quarters?

MR. TAYLOR: Yes.

MR. SPERRY: If you cut it down to ten per cent., what would happen?

MR. TAYLOR: It would take more current to build up. You must recognize that you will have to throw off current during the peak load; the current you use at that time is practically a dead loss. The current you use then simply serves to keep the furnace warm preparatory to running for the nineteen hours during the valley loads on the central station.

MR. SPERRY: What would be the efficiency running that way, compared with running twenty-four hours with the full current; would it drop six per cent. or ten per cent.?

MR. TAYLOR: That is a rather difficult question to answer.

MR. SPERRY: Of course, I understand that it is difficult to answer such a question offhand. The current that you supply on the peak load, not being sufficient to produce goods, may be a total loss so far as the production of goods is concerned.

MR. TAYLOR: If you can run the whole twenty-four hours and produce the maximum amount of goods, you are getting the full benefit. It is quite a loss to be out of the power for that length of time; at the same time I am not prepared to say that if current could be obtained at a sufficiently cheap price, both for the peak and valley load, I think in that case they would have to make the same price for the peak as the valley, because it is necessary to have it in a furnace which is to run continuously, as mine does. There may be furnaces adapted to running from twelve to nineteen hours, but my furnaces would not accomplish much in that way.

MR. SPERRY: There are many processes that do run that way.

MR. TAYLOR: There are many processes in which the electric current could be utilized in that manner.

MR. CARL HERING: The point raised by Mr. Taylor illustrates a remark I made in my paper, namely, that it may sometimes be very important to calculate the heat losses of a furnace while it is not working. In some cases that very calculation may determine whether it would be practicable or impracticable to do what Mr. Sperry suggests, and it shows, therefore, how important it is to be able to calculate these losses more correctly. The energy required for heating up a furnace is generally so great that I doubt very much whether it would pay to use furnace processes intermittently, unless this loss during the non-working period can be made very small. I should think, therefore, that the more probable field of usefulness for such spare energy would be for electrolytic processes, which can be more readily stopped.

MR. SPERRY: It is barely possible in some cases like Mr. Taylor's furnace that you might fire with gas during the peak period to keep up sufficient heat.

MR. W. E. WELLS: I do not think there is quite as fair realization of what the peak is on an electric light station. Mention has been made here of five hours. It may be that the peak scattered

through the year is on certain days five hours, but there are certain portions of the year where there is practically no peak on the electric light station, and at which time current could be furnished continuously. However, take in the winter time, from the early part of November to the latter part of December, or the early part of January, there is a decided peak every day, but the duration of that peak, taking the upper twenty-five per cent. of the load, will last all the way from five minutes at the beginning and end of this period, during the first month, to possibly an hour during the middle period, so that I think that if the electrochemical people could get together with electric light stations, arrangements might be made whereby during the greater portion of the year current could be furnished continuously. During another portion of the year current could be furnished continuously except on Saturday nights. During another and very short portion of the year, amounting to not more than forty-five days, current could be furnished continuously excepting a one-hour peak, or a peak ranging from one and a half minutes to five minutes. It may not be generally known, but the last twenty-five per cent. of the capacity of a large number of electric light stations is used only one hundred hours per year. Now, that twenty-five per cent. of capacity could be used the other 8,000 odd hours to a very good advantage.

MR. LAWRENCE ADDICKS: The various copper refining plants generally have from 5,000 to 10,000 horse-power of installation, but these small plants are very economical, running on 100 per cent. load factor, with superheated steam and good vacuum, and in these cases everything can be adjusted to uniform conditions. The price would have to be very low to be attractive, and also the process is very sensitive to changes in conditions. The tank work is a very delicate balancing of conditions, and an alteration of even 10 per cent. in current density at different times of the day would seriously disturb the silver and gold losses in the cathode.

MR. C. F. CARRIER, JR: I think there is one factor which has not been brought out in any of the discussions on either this or the previous papers, namely, that there are not two men's profits in this question. That is, there is no profit for the station man and a profit for the chemist too. I made a complete calculation

of a plant for making metallic sodium, and attempted to locate it near a central power station, and if the central power station made any money, I could not make any money, and if I made any money, the central power station could not make any money, so that it seems to me it comes down to a question of the central station running their own processes and using their own power, because two people cannot make money enough out of it. The point has been brought up by Mr. Hering, I believe, that you should calculate the loss of heat during the idle period. Now, that calculation, in my opinion, takes into consideration only a small factor. The only way you can figure is to make a complete annual balance sheet. You have to figure the cost of raw material, transportation, labor, taxes, etc., and every single item for one complete year, and every individual case must be figured separately.

MR. GRAVES: In line with that, I will say that central stations are now selling power for refrigerating purposes to be used off the peak. Now, those who are engaged in electrochemical and thermal processes are in business to manufacture and sell their products, and perhaps by-products. If a plant is introduced on the premises, the time of the high priced men in looking after the plant and watching the economical operation of it could be used to a great deal better advantage in the refining of the processes of manufacture, whereas in the case of the central station the energies of the entire force and organization are directed in making the current economically and the service reliable.

In connection with this I might mention a contract which has been made with the city by the Brooklyn Edison Co. and the New York Edison Co. for about 7,000 horse-power in Brooklyn and 15,000 in New York whereby the central station companies agree to pay the city $500 a minute for every minute beyond the first three minutes in which the current is off the line.

MR. F. A. J. FITZGERALD: The best way of determining the losses in a resistance furnace is to cut down the voltage, thus adjusting the rate of consumption of energy so as to hold the temperature constant. This can be done very well in the case of furnaces such as the induction furnace. This has been done, and it has been found in some cases that one-third of the power used in normal working will keep the temperature constant.

From this it would be easy to calculate what energy must be used during the period of peak load.

Mr. Henry D. Hibbard: No one seems to have thought of the possibility of using the valley load for mechanical purposes. Now, it is not to be thought of, probably, except in cases where the expense for power is a large part of the expense of carrying on the mechanical operation. In a machine shop, or anything of that nature, where the chief expense is labor, they must have power when they want it, but there may be something done in such lines as grinding, where the large expense of the operation is for power, and where the labor expense is low. I throw that out as a suggestion, as this idea of using the current for mechanical purposes, under the conditions suggested, seems to be as promising as any, in view of the remarks that have been made.

Mr. G. M. Kebbe: The first speaker said that he was glad the central station people had taken the initiative in bringing this thing up, and that called to my mind a remark made by you, Mr. President, and that was the correction in Mr. Meyer's paper, as to the time necessary to operate a carborundum furnace, and my point is this: Mr. Meyer is a central station man, and in presenting his paper doubtless he used the best information he had at hand, and the difficult thing in sifting this question down to a practical basis, it seems to me, is that the central station people do not understand the needs of the electrochemist, and the electrochemists apparently do not understand the possibilities of central station services.

Some one of the speakers remarked on the price of power. I do not think it is time to discuss rates, but I read recently, I think it was in the last number of Dr. Roeber's paper, that in Switzerland they are making steel in the electric furnace, and pay $70 per kilowatt year for their current for that purpose, and higher rates for the current which is used for mechanical energy, etc., in connection with the plant, and in conversation with Dr. Richards at the electrical show recently I understood him to say that they are making the steel on that basis profitably.

Now, while it is possible to obtain much lower figures on these at Niagara Falls (I just want to say that my own knowledge of this matter is like that of the other central station men—somewhat limited, and so most of my examples are hearsay), I understand

there is one plant at Niagara Falls that has a by-product of chlorine which they have a great deal of difficulty in disposing of. It would not be profitable to concentrate the chlorine in order to ship it properly, and then after it reaches its destination to dilute it again in order to put it into use, and if that same process, all other conditions being equal, was located, say, in Providence, taking power from a central station, the chlorine which is now an expensive thing to get rid of might be made a valuable by-product in the bleacheries located in Providence and Fall River and New Bedford and other eastern points. Most of the electrochemists seem to take this stand in talking of rates; they seem to consider a kilowatt year of 8,760 hours a standard. Now, there is a no-load period in the manufacture of every product using furnaces with twenty-four hours' run, or runs of shorter time, for there are certain mechanical difficulties, if not electrical difficulties, which make them necessarily intermittent. There has to be a time for certain adjustments, for re-charging, etc., and if this load factor is taken into consideration that would be one help.

Now, again, water power can probably be had at a lower rate in most instances than power from any other prime mover, but if you locate your plant at a water power and overlook the other points, the resultant figure for your energy may be higher after all. Tide water shipping facilities for the raw material, and labor which is found surrounding tide water locations, may be less, and you may have also a nearer market for your finished product.

We are willing to take the initiative in putting this thing up to you because we believe if you will consider it with us it will result to the mutual advantage of both central stations and the electrochemist.

MR. MILTON M. KOHN: It seems to me the statement given by one central station representative is of great importance in considering the matter of prices to be charged for current. My experience with most of the central station companies is that the rates they are allowed to make are controlled by public service commissions, or rates which are established by the legislature. In that case the rates are too high for the use of electric current in furnaces. I would like to know if some arrangements cannot be made by means of which central stations all over the country,

where electric furnaces can be used, could make some special terms for that class of work. It seems to me the commercial use of the electric furnace will be absolutely dependent on the special terms that the central stations can make.

MR. W. E. WELLS: Even in the State of New York, where we have several commissions, a central station can make a rate to suit the service which it is to perform. The rate fixed by the Public Service Commission of 10 cents or 12 cents a kilowatt hour is for lighting current, but where the conditions are entirely different a differential rate may be established.

MR. KOHN: As I understand it, the very best rate that the electric light companies are allowed to make is 3.5 cents, which is far too high for ordinary electric furnace work, in my opinion.

MR. WELLS: The minimum rate is not governed at all by the legislature or by the public service commission; only the maximum rate which we can charge is regulated. However, whatever minimum rate we make, that rate must be made for all, so there will be no discrimination.

MR. GEORGE BREED: New York State is not the only State in the Union. I know of a great many electric light and power companies whose minimum rate is governed entirely by the cost of coal and the cost of operating the stations; furthermore, that if the people who are interested in electrochemical manufacture will approach the managers of these stations they will be met with a perfectly open mind, and without legal restraint of any kind, will get the best rates that it is possible to give them, and they will be considerably below 3.5 cents.

MR. KOHN: I would like very much to be in touch with some of the gentlemen who will give the names of any companies, furnishing power by means of steam, which will make a rate around two cents for 75, 100, or 150 kilowatt hours per day. I would like to be in touch with some of them.

MR. BREED: Get in touch with me.

PROF J. W. RICHARDS: As to the concrete example which Mr. Kebbe brought forth, I think we can get some good ideas by considering that. In the last number of "Electro-Metallurgical Industry" there is a description of a plant in Switzerland which I visited this summer. They get power from the city power station. They run electric furnaces, one heat in the morning

and one in the afternoon. This displaced a crucible steel plant. They do not run at night, and if they did they could easily make four heats a day. They make one heat in the morning and one in the afternoon, and shut down the plant for the rest of the day, going along perhaps a little easier than we would do here in the United States; but they are many foundries in the United States which run during the day and make a heat, and do not run during the night unless they are very busy. Now, such a furnace as that takes power for about five hours in the morning and five hours in the afternoon. If they wanted to run more continuously they could perhaps take five hours after midnight. They could arrange to take another heat, so as to fit in with hours that would be satisfactory to the central station managers. I think in the case not only of electric smelting of steel, but electric smelting in brass foundries and other metal foundries, they could work only during the day and could fit in their melting hours to suit the central station.

I think there are also probably some electrolytic processes, perhaps not copper, but other electrolytic processes, refining processes, which can be absolutely stopped for a certain number of hours, not run on light load, but more easily stopped entirely for four or five hours, and then started up again without practically any hindrance at all. That is a question of whether it will pay to stop and reduce the output of a plant to so much; whether the cheaper power will pay for the restricting of their output.

I think it would be well for electrochemists, in considering different kinds of processes, to consider whether the process cannot be so modified, or practically stopped; whether the electrolytic processes cannot be stopped to fit in with the number of hours when current can be got at the cheap rate, and whether the carborundum furnaces could be arranged to run nineteen hours instead of thirty-six hours. I have no doubt a carborundum furnace could be made to run nineteen hours instead of thirty-six hours, but under the conditions as they exist at Niagara Falls it is more profitable to run them thirty-six hours.

MR. S. S. SADTLER: You might have several carborundum furnaces, some which you use for 36 hours, and other smaller ones for, say, 18 hour runs, which might not show a very extraordinary decrease in cost of production.

MR. P. MC. N. BENNIE: Looking at this question from the point of view of dollars and cents, we have done a great deal of figuring on continuous processes, taking an average amount of power, and also on absolutely discontinuous processes, and we have conferred with some central station men and have quotations for power; but we have not yet found any central station where we could apply the process as economically as we could by going to Niagara Falls and securing continuous power.

These large generating companies of Niagara Falls are also acquainted with "load factors," and they have that same problem, to a very large extent, in all of the stations, and they have been looking for the same solution right among these works and they have not found it. I think perhaps there has been a good deal of dreaming on this idea of getting an intermittent load and making it a success. There has been a great deal of work done on it, but there has not been any solution of it.

As far as manufacturing processes are concerned, when it comes down to a thing like Mr. Kohn spoke of, a small furnace, it might be practicable, but when you are speaking of the case of thousands of horse-power I do not think you will find a solution along the lines of the central station plant.

MR. LAWRENCE ADDICKS: I do not think the relative importance of the power cost in electrochemical industries to the total cost is fully understood. There is an impression that it is much the largest item. Speaking from my own experience in the copper refining business, the power runs about 20 per cent. of the cost, so that a 10 per cent. saving in power represents only 2 per cent. of the total, and other factors may easily out-balance that.

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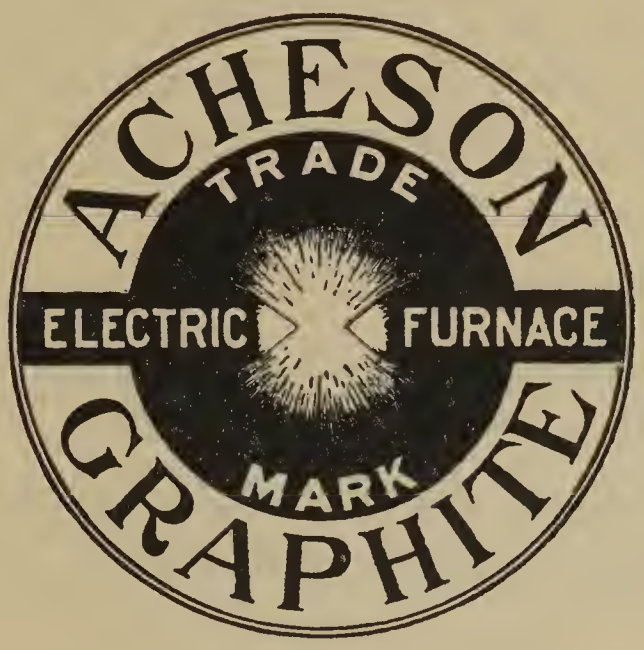

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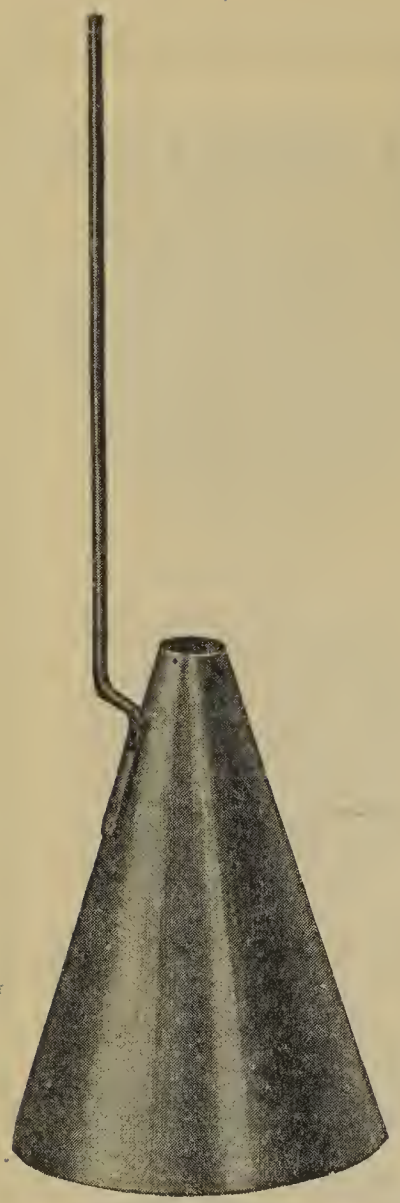

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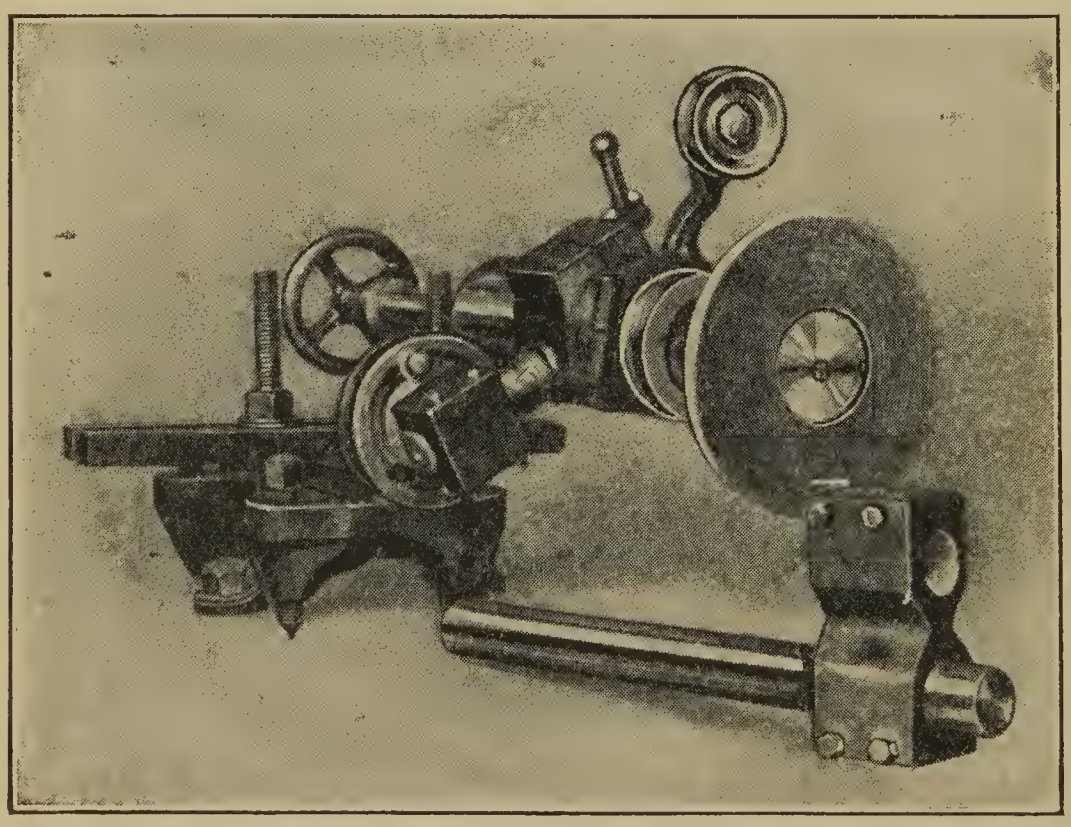

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